# REMEDIAL ACTION CONTRACT 2 IN REGION 5

# ATTACHMENT A FIELD SAMPLING PLAN FOR U.S. SMELTER AND LEAD RESIDENTIAL AREA SUPERFUND SITE EAST CHICAGO, LAKE COUNTY, INDIANA

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A-1 SITE LOCATION MAP

A-2 USS LEAD RESIDENTIAL AREA ZONES

# **Attachment**

A STANDARD OPERATING PROCEDURES

#### ACRONYMS AND ABBREVIATIONS

μm Micrometer °C Degree Celsius

ARCO Anaconda Copper Company

ASTM American Society for Testing and Materials

bgs Below ground surface

CLP Contract Laboratory Program

COC Chain of custody

CPR Cardiopulmonary resuscitation
CRL Central Regional Laboratory

dpi Dots per inch

DOT U.S. Department of Transportation
Dupont E.I. duPont de Nemours Company

EERS Emergency and Rapid Response Team EQM Environmental Quality Management

FSP Field sampling plan

HASP Health and safety plan HDPE High-density polyethylene

Hg Mercury

HRS Hazard Ranking System

ID Identification

IDEM Indiana Department of Environmental Management

IDW Investigation-derived waste ISBH Indiana State Board of Health

mg/kg milligram per kilogram

mL Milliliter
MS Matrix spike

MSD Matrix spike duplicate

NA Not applicable

NPDES National Pollutant Discharge Elimination System

NPL National Priorities List

OU Operable Unit

PAL Project action limit
PCB Polychlorinated biphenyl

PPE Personal protective equipment

PTFE Polytetrafluoroethylene

QA Quality assurance

QAPP Quality assurance project plan

QC Quality control

RAC Remedial action contract RAL Remedial Action Limit

RCRA Resource Conservation and Recovery Act

RD Remedial Design
RI Remedial Investigation
ROD Record of Decision

RSL Residential Screening Level

SAP Sampling and analysis plan

SARA Superfund Amendments and Reauthorization Act

SMO Sample Management Office SOP Standard operating procedure

SOW Statement of work

START Superfund Technical Assessment and Response Team

SVOC Semivolatile organic compound

TAL Target Analyte List
TC Toxicity characteristics

TCLP Toxicity characteristics leaching criteria

TCRA Time Critical Removal Action
TSCA Toxic Substance and Control Act

USCS Unified Soil Classification System USDA U.S. Department of Agriculture

U.S. EPA U.S. Environmental Protection Agency

USS Lead U.S. Smelter and Lead Refinery Superfund Site

VOC Volatile organic compound

WA Work assignment
Weston Weston Solutions, Inc

XRF X-ray fluorescence

#### A1.0 INTRODUCTION

Under the U.S. Environmental Protection Agency (U.S. EPA) Remedial Action Contract (RAC) 2 for Region 5, Contract No. EP-S5-06-02, Work Assignment (WA) No. 198-RDRD-053J, SulTRAC has prepared this field sampling plan (FSP) as part of the sampling and analysis plan (SAP) for the USS Lead Superfund Site in Lake County, Indiana (see Figure A-1). The SAP consists of this FSP (Appendix A) and the quality assurance project plan (QAPP) (Appendix B) (SulTRAC 2014c), which are among the site-specific plans to be prepared under the WA in accordance with Task 1 of the U.S. EPA statement of work (SOW) (EPA 2014). The QAPP discusses quality assurance (QA) and quality control (QC) protocols associated with sampling and analysis activities at the USS Lead Superfund Site.

This FSP describes sampling activities SulTRAC will perform during the Remedial Design (RD) sampling in the residential area known as OU1 at the USS Lead superfund site. As outlined in the USS Lead site work plan (SulTRAC 2014a), the scope of this FSP was developed based on interaction with the U.S. EPA and review of site-related documents. FSP field investigation activities will focus on the following activities and areas:

Characterize the lateral and vertical extent of soils contaminated by lead and/or arsenic at residences, schools, parks, vacant lots, and other areas where residents and/or on-site workers may come into contact with contaminated soil. Field investigation techniques will utilize X-ray fluorescence (XRF) measurements supplemented by laboratory analyses.
 Identify residential properties (including schools and parks) where lead concentrations in soil exceed the project action limit (PAL) of 340 mg/kg and/or arsenic concentrations exceed the PAL of 26 mg/kg. PALs and Cleanup Goals (CG) are discussed further in Section A3.0.
 Identify industrial/commercial properties where lead concentrations in soil exceed the PAL of 670 mg/kg and/or arsenic concentrations exceed the PAL of 26 mg/kg.
 Evaluate whether soils contaminated by lead and/or arsenic require disposal as characteristic

#### This FSP discusses

hazardous waste.

the site background (Section A2.0),
project objectives (Section A3.0),
field sampling activities (Section A4.0),
field sampling procedures (Section A5.0),
laboratory analytical methods (Section A6.0),
decontamination procedures (Section A7.0),
sample handling procedures (Section A8.0),
disposal of investigation-derived waste (IDW) (Section A9.0),

health and safety procedures (Section A10.0),
QA/QC requirements (Section A11.0).
Section A12.0 lists references used to prepare this FSP.

Tables are included in the body of the text. Figures are provided after Section A12.0. Standard operating procedures (SOP) are provided in Attachment A.

#### A2.0 SITE BACKGROUND

This section discusses the site description (Section A2.1), site history (Section A2.2), previous investigations (Section A2.3), and current status (Section A2.4).

#### **A2.1 SITE DESCRIPTION**

The USS Lead Superfund Site is located approximately 18 miles southeast of Chicago, Illinois, in East Chicago, Indiana (Figure A-1). East Chicago is surrounded by one of the most heavily industrialized areas in the U.S., including steel mills, oil refineries, heavy manufacturing, chemical processing plants, and heavy rail. The Residential Area (OU1) of the USS Lead Superfund Site is primarily a low-income residential area with commercial and light industrial areas nearby.

The East Chicago area has historically supported a variety of industries. In addition to the USS Lead smelting operation, some other industrial operations may have also managed lead and other metals. For example, immediately east of the former USS Lead Facility (OU2), across Kennedy Avenue, is the former DuPont site (currently leased and operated by W.R. Grace & Co., Grace Davison). One of the processes that historically took place at the DuPont site was the manufacturing of the pesticide lead arsenate. On the western edge of OU1, west of Gladiola Street and north of 151st Street, two smelter operations reportedly managed lead and other metals. A 1930 Sanborn Fire Insurance Map identifies the operations as Anaconda Lead Products which was a manufacturer of white lead and zinc oxide and International Lead Refining Company which was a metal-refining facility.

#### A2.2 SITE HISTORY

The USS Lead Superfund Site is comprised of two Operable Units (OU) (Figure A-1). OU2 is the U.S. Smelter and Lead Refinery, Inc. facility and is located on a 79-acre tract of land in East Chicago, Indiana and includes site wide groundwater. OU1 is made up of nearby commercial, municipal, and residential properties north of OU2 (Figure A-1). The contaminants of concern at the site are lead and arsenic. Land use within the OU1 boundaries is a mix of residential, commercial, and industrial properties. The area also includes schools, churches, playgrounds, parks, and small businesses. While USS Lead was a significant contributor to contamination in the residential area, EPA's investigations indicate that other facilities in the area have also been significant sources of contamination to the residential area.

The USS Lead facility (OU2) is a former lead smelter located at 5300 Kennedy Avenue, East Chicago, Indiana. In 1920, the property at OU2 was purchased by U.S. Smelting, Refining, and Mining, and later

by USS Lead. At that time, USS Lead operated a primary lead smelter at the facility. Between 1972 and 1973, the facility at OU2 was converted to a secondary lead smelter, which recovered lead from scrap metal and automotive batteries. All operations at OU2 were discontinued in 1985. Two primary waste materials were generated as a result of the smelting operations: (1) blast-furnace slag and (2) lead-containing dust emitted from the blast furnace stack. (SulTRAC 2012a).

In September 1985, the Indiana State Board of Health (ISBH) found USS Lead in violation of State law because lead particles were found downwind of the site in the residential area north of the facility (OU1). Approximately four million people draw drinking water from intakes primarily into Lake Michigan, which is 15 miles downstream of where hazardous substances from the site enter surface water. Seventy-five hundred people work or attend school within two miles of the site (SulTRAC 2012a).

As part of a Resource Conservation and Recovery Act (RCRA) Corrective Action in 2003 and 2006, EPA conducted soil sampling in OU1. In the 2003 EPA RCRA investigation, eighty-three residential properties within OU1 were sampled and analyzed for lead using a Niton XRF instrument in late July and early August 2003. Soils from 43 locations (52 percent) exceeded the 400 mg/kg residential soil screening criterion for lead. On January 22, 2008, EPA approved a time-critical removal action for private residential properties within OU1 due to elevated levels of lead in surface soils identified during investigations conducted from 2002 through 2007. EPA identified 15 private properties that contained soil with lead concentrations exceeding the "regulatory removal action level" of 1,200 mg/kg in the top 6 inches of soil. EPA was able to obtain access agreements to only 13 of the 15 properties. The properties were remediated between June 9 and September 22, 2008, by Weston Solutions, Inc. (Weston) and Environmental Quality Management (EQM) under a Time Critical Removal Action (TCRA). The properties were excavated to a depth of 1 to 2.5 feet below ground surface (bgs). Weston used an XRF instrument to field screen and confirm that excavation was completed to a depth where lead concentrations were below 400 mg/kg. All the properties were backfilled with clean fill and re-sodded by September 25, 2008. A total of 1,838 tons of soil was transported offsite to a landfill facility as special waste (SulTRAC 2012a).

The USS Lead Superfund Site (comprising both OU1 and OU2) was evaluated under the Hazard Ranking System (HRS) in September 2008 and was found to have an observed release of lead in the air migration pathway as well as the surface water migration pathway (U.S. EPA 2008). The USS Lead Site was listed as a Superfund site on the National Priorities List (NPL) on April 8, 2009.

#### A2.3 PREVIOUS SITE INVESTIGATIONS AND REMOVAL ACTIONS

Many investigations have been conducted as part of the USS Lead Superfund Site (OU1 and OU2). The following is a list of the investigations that have been conducted from 1985 to the present. These investigations are discussed in the Remedial Investigation (RI) report (SulTRAC 2012a).

	1985 Inspection Report of Hammond Lead and USS Lead Refining Soil Survey (OU2)
	2001 Site-Wide Sampling and Analysis Report (OU2)
	2001 USS Lead MRFI Addendum Off-Site Sampling and Analysis Report (OU2)
	2002 Air Dispersion Modeling and Historical Aerial Photography Review (OU1 and OU2)
***************************************	2003 Report on X-Ray Fluorescence Field Study of Selected Properties in Vicinity of Former USS Lead Refinery Facility, East Chicago, Indiana (OU1)
	2004 Off-Site Soil Excavation, Howard Industries (OU2 HI Triangle Area)
	2004 Off-Site Soil Excavation, Indiana Harbor Belt Railroad (OU2 IHBRR Triangle Area)
	2004 Off-Site Soil Excavation Kennedy Avenue (OU2 Eastern Off-Site Area)
	2004 On-Site Soil Excavation (OU2 Wetlands Area)
	2004 Draft Final USS Lead, Modified RCRA Facility Investigation (MRFI) Report (OU2)
Name of the last o	2004 Draft Characterization of Lead and Other Metals in Soil in the Vicinity of the USS Lead Site, East Chicago, Indiana (OU1 and OU2)
	2006 EPA FIELDS Investigation (OU1)
	2007 STN Draft Site Assessment Letter Report, USS Lead Site (Background Study)
	2008 Hazard Ranking Summary Documentation Record (OU1 and OU2)

# 2008 CERCLA Time Critical Removal Action:

On June 9, 2008, U.S. EPA; Weston, Superfund Technical Assessment and Response Team (START); and Environmental Quality Management (EQM), the Emergency and Rapid Response Services (ERRS) contractor, mobilized to the Site to begin the removal of lead-contaminated soil from 13 residential properties. The residential removal cleanup level was established at 400 mg/kg.

Removal activities were completed on September 25, 2008. The ERRS contractor arranged for the transportation and disposal of 1,838 tons of lead-contaminated soil at the Forest Lawn Landfill located in

Three Oaks, Michigan. Clean backfill for the Removal Action totaled 3,120 cubic yards (U.S. EPA 2009).

#### 2011 Time Critical Removal Action:

EPA completed a time critical removal action (TCRA) of 16 properties with lead in soil concentrations exceeding 400 mg/kg from October through December 2011. The TCRA consisted of removing lead-contaminated soil from five East Chicago public housing addresses and 11 residential properties (two of which were not remediated in the prior 2008 TCRA due to access issues). The TCRA was conducted between October 24 and December 9, 2011. Approximately 1,913 tons of low-level lead-contaminated soil were excavated during the TCRA and the material was sent to an off-site location for disposal. Each property was backfilled to the existing grade and seeded after the soil removal was completed.

# 2009 Remedial Investigation/Feasibility Study:

In 2009, U.S. EPA Superfund began a Remedial Investigation and Feasibility study. In 2010, the City of East Chicago remediated yards at two properties that were above the U.S. EPA residential screening level (RSL) for lead (400 mg/kg) The Remedial Investigation and Feasibility Study were completed in 2012 (SulTRAC 2012a; SulTRAC 2012b).

#### A2.4 CURRENT SITE STATUS

On November 30, 2012, EPA signed a Record of Decision (ROD) detailing the planned cleanup for OU1. The selected remedy involves removing impacted soil that exceeds RALs, to a maximum excavation depth of 2 feet, but leaving remaining soils below 2 feet in place. This alternative requires excavation of soil exceeding RALs, disposal of excavated soil at an off-site Subtitle D landfill, and, as necessary, treatment of some soil that may exceed the toxicity characteristic (TC) regulatory threshold to meet land disposal restrictions. EPA estimates that soil with lead concentrations above 2,600 mg/kg (an estimated 7% of the excavated yards at OU1) exceeds the TC regulatory threshold toxicity characteristic based on toxicity characteristic leaching criteria (TCLP) testing conducted during the RI. Soil exceeding RALs would be excavated to a depth determined by pre-remedial sampling results. The maximum excavation depth is estimated to be 24 inches, but actual excavation depth at each property will vary based on results of the Remedial Design sampling. Only soil with lead or arsenic concentrations above the PALs will be excavated. Excavated soil will be replaced with clean backfill, including 6 inches of top soil, to maintain the original grade. Each yard will be restored to its pre-remedial condition. If contaminated soil is identified at a depth greater than 24 inches bgs, a visual barrier, such as orange construction fencing or landscape fabric, will be placed above the contaminated soil and beneath the clean backfill soil. Institutional controls will be implemented to prevent exposure to contaminated soils beneath the barrier.

#### A3.0 PROJECT OBJECTIVES

The purpose of this FSP is to describe the approach that will be used to conduct the remedial design (RD) sampling at OU1. The objective of the RD sampling is to characterize the lateral and vertical extent of lead-contaminated and arsenic-contaminated soils at residences, schools, parks, vacant lots, and other areas where residents or workers may come into contact with contaminated soil within OU1. The data obtained from this RD sampling program will be used to develop remedial design documents for future remediation of properties needing soil removal to reduce risks to human health.

OU1 has been divided into three zones as shown on (Figure A-2). Zone 1 is the Public Housing area east of McCook Avenue and includes the Carrie Gosch Elementary School. Zone 2 is the residential/commercial area east of the Public Housing Area and west of the Elgin Joliet and Eastern Railway. Zone 2 also includes the properties along Vernon Avenue to the North of Carrie Gosch School. Zone 3 includes the residential/commercial area east of the Elgin Joliet and Eastern Railway. The Remedial Design sampling will be conducted in multiple phases based on which zone is being sampled. SulTRAC anticipates that Zone 1 will be sampled will be sampled in one continuous event followed by Zone 3, which will likely be sampled in two sampling events based on availability of access agreements from property owners. SulTRAC anticipates that Zone 2 will be sampled last in two to three sampling events based on property access. SulTRAC intends to conduct RD sampling in each zone in an uninterrupted process to the extent possible. However, it is anticipated that some properties will lag behind due to difficulties obtaining access agreements and SulTRAC will be required to go back to a particular zone to complete sampling.

All SulTRAC field activities will be conducted in accordance with the U.S. EPA-approved, site-specific QAPP (Appendix B) (SulTRAC 2014c) and SulTRAC SOPs (see Attachment A). Where this FSP differs from the SOPs, the FSP's site-specific procedures will take precedence.

#### A3.1 CLEANUP GOALS AND PROJECT ACTION LIMITS

A central component of EPA's alternatives evaluation during the RI/FS process is the development of CGs for the response action that are protective of human health and the environment. Numerical CGs for the USS Lead Superfund Site are based on risk calculations performed during the RI that are protective of human health and the environment (SulTRAC 2012a).

PALs are the concentrations at which a decision will be made. The PALs for the USS Lead Superfund Site were developed from the risk based CGs developed during the RI/FS process (SulTRAC 2012a).

PALs incorporate uncertainty in the analytical data based on statistical evaluations of precision, accuracy, and bias observed in the RI data. Combining the risk based CG with a margin of error (analytical uncertainty) results in PALs which are lower than the CGs to prevent false negative conditions (type II error) where a property would be characterized as clean when in fact the CGs have been exceeded.

The USS Lead Superfund Site is located approximately 18 miles southeast of Chicago, Illinois, in East Cleanup goals and PALs are shown in Table A-1. The cleanup goals were specified in the ROD that was signed in November of 2012. The PALs for lead include a safety margin for the difference between field XRF measurements and total lead analysis based on the regression analysis discussed in the RI (SulTRAC 2012a).

TABLE A-1 CLEANUP GOALS AND PROJECT ACTION LIMITS

Parameter	Cleanup Goal (mg/kg)	Project Action Limit (mg/kg)
Lead (Residential)	400	340
Lead (Industrial/Commercial)	800	670
Arsenic	26	26

Notes:

mg/Kg milligrams per kilogram

# A4.0 FIELD SAMPLING APPROACH

The field sampling approach discussed in this section pertains to the SulTRAC Remedial Design sampling. Section A5.0 provides a detailed discussion of sample collection procedures.

As discussed with the U.S. EPA and outlined in the work plan (SulTRAC 2014a), SulTRAC will conduct RD sampling activities in multiple phases based on the zone being sampled (Figure A-2). Sampling will be conducted using XRF investigations at approximately 1,150 residential properties. Further, the RD investigation will collect samples from the XRF locations to identify properties where it is expected that soils will need to be disposed of as hazardous waste or treated prior to disposal. This data will be used to further refine the correlation between XRF data and total metals concentrations for arsenic and lead. Access agreements to sample and, if necessary, remediate residential and commercial/industrial properties or properties owned by the City of East Chicago will need to be obtained for every property within the OU1 boundary.

During the RI sampling effort, 100% of the samples collected were analyzed for lead by either XRF or both XRF and CLP fixed laboratory analysis. However, only a portion of the samples were analyzed for arsenic (roughly 15% of the samples) (SulTRAC 2014a). As a result, properties that were sampled during the RI as having soil concentrations for lead below 400 mg/kg will be resampled for arsenic. Furthermore, if one or more intervals in a yard had lead concentrations above 400 mg/kg, only the deeper intervals below the last interval above 400 mg/kg will be required for arsenic testing. If all four depth intervals in a yard are above 400 mg/kg for lead, no retesting for arsenic will be required as the yard will be designated for excavation regardless of the arsenic concentration.

SulTRAC will submit soil samples with XRF readings for lead between 300 mg/kg and 400 mg/kg to a Contract Laboratory Program (CLP) laboratory for lead analysis to further refine the correlation between the XRF concentrations and CLP analytical results. SulTRAC will submit soil samples with XRF readings for arsenic between 20 mg/kg and 30 mg/kg to a CLP laboratory for arsenic analysis to further refine the correlation between the XRF concentrations and CLP analytical results. Resulting data will be used to make decisions regarding remedial actions at residential properties where XRF lead concentrations exceed the PAL of 340 mg/kg; industrial/commercial properties where XRF lead concentrations exceed the PAL of 670 mg/kg; and where XRF arsenic concentrations exceed the PAL of 26 mg/kg (as will be described in the Remedial Design document). The PALs for lead are based on the conclusions of the RI that recommended a clean-up objective equal to the EPA RSL for lead of 400 mg/kg on residential properties and 800 mg/kg on industrial/commercial properties. A safety margin has

been included in the PAL to compensate for the difference between XRF measurements and total lead results as recommended by the RI. The PAL for arsenic has been set at the clean-up goal recommended in the RI of 26 mg/kg.

If soil XRF readings indicate soil lead concentrations are greater than 2000 mg/kg, soil samples will also be collected for TCLP lead analysis through the CLP program to address soil exceeding the TC regulatory threshold for lead. If soil XRF readings indicate soil arsenic concentrations greater than 100 mg/kg, soil samples will also be collected for TCLP arsenic analysis through the CLP program to address soil exceeding the TC regulatory threshold for arsenic. If petroleum staining is visible in a soil sample, that sample will be submitted to a CLP laboratory for TCLP VOCs, TCLP SVOCs, Ignitibility, and Corrosivity.

SulTRAC will analyze samples from the 24 to 30 inch bgs interval if the native sand horizon is not encountered in the 18 to 24 inch sampling interval. If the soil below 24 inches is above the PAL, the design will require a visual barrier to be put in place to designate that underlying soils are contaminated before backfill is emplaced.

The following sections discuss the sampling approaches to be used for the Remedial Design.

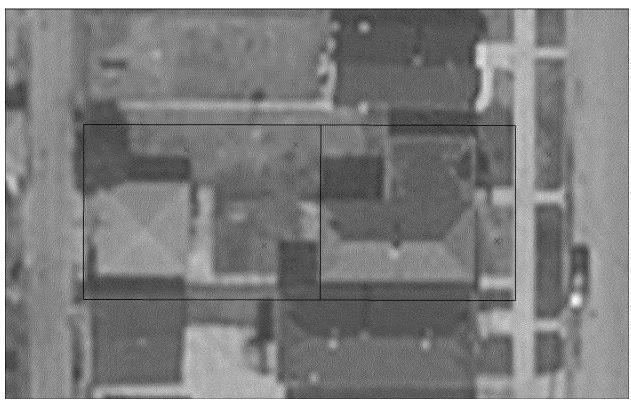
### A4.1 REMEDIAL DESIGN SAMPLING

The remedial design sampling will include the collection of XRF soil samples on each property in OU1 that does not already have sufficient data to make remedial design decisions. Eighty-nine properties were sampled during the RI and thirty-one properties have been remediated by START and the City of East Chicago. SulTRAC will collect samples from approximately 1,150 properties within OU1. The field team will attempt to distribute the sample points to provide even coverage of the property/yard sampled. SulTRAC will mobilize to OU1 to conduct an XRF soil investigation for metals. The goal of the screening is to identify residential properties above the cleanup goals for lead and arsenic and provide data adequate to prepare remedial designs.

#### **A4.1.1** Residential Properties

Residential properties with a structure on the property will be divided into front and back yards, and a 5-point composite sample will be collected from each front yard and each back yard by depth interval. Drip zones will not be sampled separately. If there are side yards, the 5-point composite may include locations from the side yards. Four depth-discrete 5-point composite samples will be collected from each

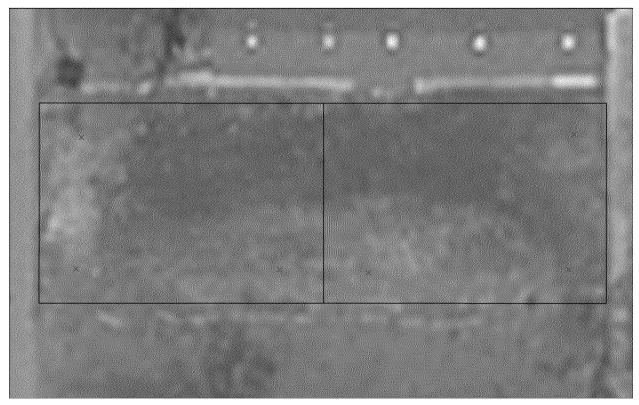
yard, including 5-point composite samples from 0-6 inches, 6-12 inches, 12-18 inches and 18-24 inches bgs, in an X-shaped pattern, with one sample aliquot from each end point of the X and one sample aliquot from the center, as shown in the figure located after this paragraph. The five individual aliquots from each point will then be mixed into a depth specific composite sample for analysis by XRF. Typically, eight composite samples will be collected at each residential property (four from the front yard and four from the back yard). If native sand is not encountered in the 18-24 inches bgs sampling interval, an additional depth interval composite sample will be collected from 24-30 inches bgs for analysis of lead and arsenic. Any composite sample from a yard with XRF readings for lead between 300 mg/kg and 400 mg/kg and/or XRF readings for arsenic between 20 mg/kg and 30 mg/kg will be submitted to CLP for total lead and/or total arsenic analysis. The Any composite sample from a yard with XRF readings for lead above 2000 ppm or arsenic above 100 ppm will be submitted to CLP for TCLP metals (lead and/or arsenic) analysis. If petroleum staining is visible in a soil sample, that sample will be submitted to a subcontract laboratory for TCLP VOCs, TCLP SVOCs, Ignitibility, and Corrosivity.



EXAMPLE COMPOSITE SAMPLE LOCATIONS - RESIDENTIAL PROPERTY

#### A4.1.2 Vacant Lots

Vacant lots will be divided into two halves to correspond with front and back yards. Four 5-point composites will be collected from each half, and each composite sample will be screened using XRF. If native sand is not encountered in the 18-24 inches bgs sampling interval, an additional depth interval composite sample will be collected from 24-30 inches bgs for analysis of lead and arsenic. Each yard will be sampled in the same manner as described above for residential properties.

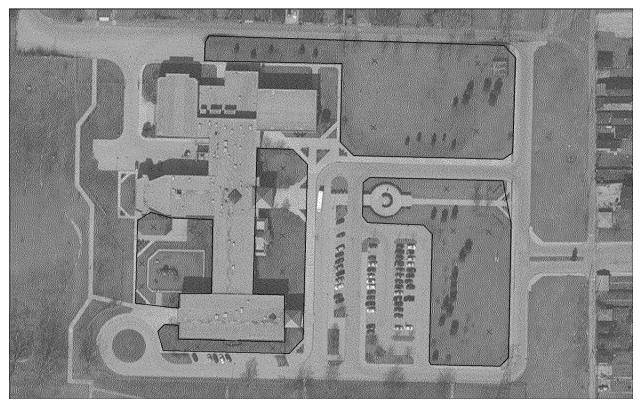


**EXAMPLE COMPOSITE SAMPLE LOCATIONS - VACANT LOT** 

#### A4.1.3 Schools

If sampling at schools becomes necessary, school properties will be sampled by dividing the property into four quadrants. If native sand is not encountered in the 18-24 inches bgs sampling interval, an additional depth interval composite sample will be collected from 24-30 inches bgs for analysis of lead and arsenic. Each quadrant will be sampled in the same manner as described above for residential properties.

#### EXAMPLE COMPOSITE SAMPLE LOCATIONS - SCHOOL



#### A4.1.4 Parks

The known parks within OU1 were sampled as part of the RI. If sampling in recreational parks is necessary, parks will be sampled by dividing the property into four quadrants of approximately equal area. If native sand is not encountered in the 18-24 inches bgs sampling interval, an additional depth interval composite sample will be collected from 24-30 inches bgs for analysis of lead and arsenic. Each quadrant will be sampled in the same manner as described above for residential properties.

# EXAMPLE COMPOSITE SAMPLE LOCATIONS - PARK 1



# EXAMPLE COMPOSITE SAMPLE LOCATIONS - PARK 2



A-15

# A4.1.5 Industrial/Commercial properties and Right-of-Ways

Industrial/Commercial properties and Right-of-Ways will be sampled by dividing the property into four quadrants of approximately equal area. Sampling will only be conducted in grassy areas or areas with exposed soil. No samples will be collected beneath paved areas or areas covered by concrete. If native sand is not encountered in the 18-24 inches bgs sampling interval, an additional depth interval composite sample will be collected from 24-30 inches bgs for analysis of lead and arsenic. Each quadrant will be sampled in the same manner as described above for residential properties. Any composite sample from a quadrant with XRF readings for lead between 650 mg/kg and 800 mg/kg and/or XRF readings for arsenic between 20 mg/kg and 30 mg/kg will be submitted to CLP for total lead and/or total arsenic analysis.



EXAMPLE COMPOSITE SAMPLE LOCATIONS - COMMERCIAL/INDUSTRIAL

# EXAMPLE COMPOSITE SAMPLE LOCATIONS - RIGHT OF WAY



A-17

SulTRAC will sample approximately 1,150 properties as part of the Remedial Design sampling. Samples will be analyzed using appropriate EPA methods for all chemical analysis as identified in Section A6.0 of this FSP.

#### A5.0 FIELD SAMPLING PROCEDURES

This section describes the procedures to be used in the collection of samples described in Section A4.0. Specifically, this section details the procedures and methods that will be used to collect Remedial Design samples.

As discussed in Section A4.1.1, a soil investigation for arsenic and lead will be conducted on properties in the OU1 residential area site. SulTRAC will collect samples from properties within OU1 that do not have sufficient data for remedial decisions and design data.

Soil composition at each soil screening location will be documented in the field notebook. The following information will be recorded for each soil sampling location: location number, date completed, time, field personnel's initials, and location sketch with a north directional arrow (with adequate information to locate the individual locations for each component of the 5-point composite sample). The lithologic description will also be recorded for every location and will include color, texture, and lithology. If slag or stamp sands are encountered, this information will be clearly identified in the field notebook. All soil screening sample identification (ID) numbers (Section A8.2) will be entered in the field log book. All composite soil-screening locations will be photographed with the sample ID number written on a whiteboard and a geographic landmark of some kind in the field of view. Photographs will be archived.

Five-point composite samples will be collected from each yard or quadrant of each selected property in the configuration described in Section 4.1. Samples will be collected from 0 to 6 inches, 6 to 12 inches, 12 to 18 inches, and 18 to 24 inches bgs, as recommended in the Superfund Lead-Contaminated Residential Sites Handbook (EPA 2003). If native sand is not encountered in the 18-24 inches bgs sampling interval, an additional depth interval composite sample will be collected from 24-30 inches bgs for analysis of lead and arsenic. Samples will be collected with a 6-inch-long by 3-inch-diameter bucket auger or shovel, which will be advanced below the ground surface at each location comprising the 5-point composite sample. One aliquot of soil from each depth at each location will be placed in a separate one-gallon Ziploc<sup>TM</sup> plastic re-sealable bag, with one bag dedicated to each sample depth. Each bag will be transported back to the SulTRAC field office where it will be thoroughly composited by shaking and stirring, the bag will be analyzed via XRF, and the results recorded in the XRF logbook. Data logging of XRF readings will also be performed and sample results will be transmitted to the data manager daily. If concentrations meet criteria described above, samples for CLP laboratory analysis will be collected from the same bag. Any excess soil not used for sampling will be used to fill the boring locations. If necessary, auger borings will be filled to the top with potting soil so that no hole remains in the areas sampled.

Use of the XRF method is restricted to personnel trained and knowledgeable in the operation of an XRF instrument. For measurement, the soil sample is placed in a plastic bag, positioned in front of the probe window, and measured. The probe window is placed in direct contact with the plastic bag, mainly to preserve the XRF window quality (see SOP XRF in Attachment A).

Soil samples with lead results between 300 mg/kg and 400 mg/kg or arsenic results between 20 mg/kg and 30 mg/kg will be sent to a CLP laboratory for analysis of lead and/or arsenic. Soil samples with lead above 2000 mg/kg or arsenic concentrations above 100 mg/kg will be sent to a CLP laboratory for analysis of TCLP lead and/or arsenic. If petroleum staining is visible in a soil sample, that sample will be submitted to a CLP laboratory for TCLP VOCs, TCLP SVOCs, Ignitibility, and Corrosivity.

All samples for CLP analysis will be placed in an iced sample cooler and maintained at a temperature of  $4 \pm 2$  °C without freezing until delivery to the laboratory under standard chain-of-custody (COC) protocol.

#### A6.0 LABORATORY ANALYTICAL METHODS

Table A-2 lists the laboratory analytical methods for the samples collected by SulTRAC. Field investigation samples for total metals and TCLP metals will be analyzed by the CLP laboratory. TCLP VOCs, TCLP SVOCs, Ignitibility, and soil pH will be analyzed by the subcontract laboratory (CT Laboratories).

TABLE A-2 ANALYTICAL METHODS SUMMARY

Parameter	Laboratory	Analytical Method <sup>a</sup>
Metals (arsenic/lead by XRF)	CLP	EPA Method 6200
Metals (arsenic/lead)	CLP	CLP SOW ISM01.3
TCLP lead and arsenic	CLP	CLP SOW ISM01.3MA
TCLP VOCs	CT Laboratories	EPA Method 1311/8260C
TCLP SVOCs	CT Laboratories	EPA Method 1311/8270C
Ignitibility	CT Laboratories	EPA Method 1010A
pH	CT Laboratories	EPA Method 9045D

#### Notes:

CLP Contract Laboratory Program

MA Modified Analysis SOW Statement of work

SVOC Semivolatile organic compound

TAL Target Analyte List

TCLP Toxicity characteristic leaching procedure

VOC Volatile organic compound

a EPA 2010; see Section A12.0

#### A7.0 DECONTAMINATION PROCEDURES

Sampling equipment will be decontaminated following the general practices detailed in SOP 002. Disposable sampling equipment will be used to collect individual grab samples only, and will be discarded rather than decontaminated. Soil samples will be collected with hand augers or shovels. The augers/shovels will be washed with a brush and non-phosphate detergent (such as Alconox), then washed and thoroughly rinsed with potable water. To prevent cross contamination, measuring and sampling equipment will be decontaminated prior to the initiation of sample collection activities and between each consecutive sampling location. Wash water will be disposed of in the yard where soil collection occurred and will not be containerized.

#### A8.0 SAMPLE HANDLING PROCEDURES

SulTRAC will collect soil samples for analysis at a CLP Laboratory; prepare the samples for shipment; complete all necessary documentation; and decontaminate non-disposable equipment. The following sections discuss sample containers, preservatives, and holding times; sample ID; sample documentation; sample COC; and sample packing and shipment. Samples for XRF analysis will be maintained in the custody of the field sampling team or XRF technician at all times and will not be shipped to an offsite laboratory.

#### A8.1 SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

SulTRAC anticipates collecting soil samples. Sample handling and procedures are different for each type of chemical group analysis and matrix type. Table A-3 summarizes sample container, preservation requirements, and holding-time requirements for this project.

TABLE A-3 SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

Matrix	Analyte	Sample Container	Preservation Requirements	Maximum Holding Time <sup>a</sup>
Soil	XRF Metals (arsenic and lead)	1 gallon plastic bags	None	6 months
Soil	Metals (arsenic and lead)	Two 4-ounce wide-mouth glass jar(s)	Cool to 4 ± 2 °C	6 months
Soil	TCLP (arsenic and lead)	Two 4-ounce wide-mouth glass jar(s)	Cool to $4 \pm 2$ °C	6 months
Soil	TCLP VOCs	16-ounce wide-mouth glass jar	Cool to 4 ± 2 °C	14 days to extraction/ 14 days to analysis
Soil	TCLP SVOCs	Use 16-oz jar above	Cool to $4 \pm 2$ °C	14/7/40 <sup>b</sup>
Soil	Ignitibility	Use 16-oz jar above	Cool to $4 \pm 2$ °C	ASAP
Soil	Soil pH	Use 16-oz jar above	Cool to $4 \pm 2$ °C	ASAP

Notes:

a Holding time is measured from the time of sample collection to the time of sample extraction and analysis (EPA 2004)

b 14 days to TCLP extraction, 7 days to SVOC extraction, 40 days from extraction to analysis

<sup>°</sup>C Degree Celsius

#### A8.2 SAMPLE IDENTIFICATION

Properties will be identified using a unique sequential sample ID number. The identifier will have the following format:

Street – sequential number – yard/quadrant – depth – sample type

Sample identifiers will consist of the first three letters or numbers of a street name (e.g., DRU for Drummond, 151 for 151<sup>st</sup> Street); a sequential number will follow (e.g., "1000" for the first property sampled); a yard or quadrant designator ("F" for front yard facing street, "B" for back yard, and "NW, NE, SE, or SW" for quadrants); a depth designator ("0 – 6" for zero to 6 inches bgs). For example, a sample collected from 12 to 18 inches bgs in the back yard at 4856 Drummond Street which is the 231st sample collected by the sample team would be designated as DRU231-B-12-18. The sample date and time will be recorded in field notebooks and on chains-of-custody forms. Sample team 1 will begin sequential numbering at "1000" and sampling team 2 will begin their sequential numbering at "5000" to prevent duplicating sample numbers.

CLP Scribe<sup>TM</sup> software will also assign each laboratory sample an ID number. Scribe was developed to expedite sample documentation, track samples from the field to the laboratory, and reduce the most common documentation issues associated with sampling. Before or during the sampling event, the user will enter information regarding the site, project, sampling team, analysis, location, matrix, collection time and date, and sample and tag numbers.

#### A8.3 SAMPLE LABELS

Scribe generates labels for samples sent to a CLP Laboratory. A sample label will be affixed to each sample container being shipped to a CLP Laboratory. The label will be completed with the following information:

Project number
CLP case number
CLP sample number
Sample station name (sample ID number)
Sample collection date and time
Preservative
Sample collector's initials

Analysis required
Sample tag number

After labeling, each sample will be preserved as required (see Table A-3).

#### A8.4 SAMPLE DOCUMENTATION

Sampling activities will be documented in a field notebook using an ink pen. At the start of each day, the following information will be noted: weather, site conditions, field staff present, subcontractors present, and any meetings conducted. The field team will record the following information in the field logbook for every sample: collection time, sample ID number (not CLP ID number), sampling depth, sampling location description, field observations, sampler's name, time of sample collection, and analyses. Field log books will document the locations of each sample point and hand drawn locations will be included on the sampling map.

Each page of the field notebook will be dated and numbered (if appropriate); and each day's notes will be signed by SulTRAC personnel. Any residual space on the last page of each day's log will be crossed out with a single line. Each new sampling day shall begin on a new page in the field notebook. Any corrections made during the same day of sampling will be crossed out with one single line, or the term "backnote" can be inserted to account for missed time.

The field team leader will ensure that all documentation in the field notebook is appropriately recorded. Any corrections or additions can be made on a subsequent page with appropriate documentation, although this approach is not recommended, and corrections or additions are best made the same day as the sampling.

All field notebooks must be kept secure by the field team leader during the field work period. As possible, all field notebooks shall be scanned electronically at high resolution (minimum 300 by 300 dots per inch [dpi]). If, after 1 week of continuous field work, field notes cannot be electronically scanned, high-resolution hard copies must be made and kept secure until electronic scanning can be performed. All completed field notebooks and any hard copies will be stored with the project manager in the Chicago office. Field data records will be maintained in accordance with EPA's "Multi-Media Investigation Manual" (EPA 1992b) and this FSP.

#### A8.5 SAMPLE CHAIN OF CUSTODY

SulTRAC will use standard sample COC procedures to maintain and document sample integrity for

accord	ance with the SulTRAC RAC 2 Contract Level QAPP. A sample will be considered in custody if
one of	the following statements applies:
	It is in a person's physical possession or view.
	It is in a secure area with restricted access.
	It is placed in a container and secured with an official seal so that the sample cannot be reached without breaking the seal.
For sar	mples to be sent to a CLP Laboratory, Scribe generates and prints COC forms, called traffic reports
(a labo	ratory copy and a region copy). The laboratory copy will be sealed inside the lid of the sample
shipme	ent container. COC procedures provide an accurate written record that traces the possession of
individ	lual samples from the time of collection in the field to the time of acceptance at the laboratory. One
COC r	ecord will be generated for each container shipped. The COC record also will be used to document
	uples collected for shipment to a CLP Laboraotry and the analyses requested. The following
	ation will be documented on the COC form:
111101111	
	Project name and number (region copy only)
	CLP or CRL case number
	CLP or CRL sample numbers
	Sample tag numbers
	Sampling location (station ID number)
	Name and signature of sampler
	Destination of samples (laboratory name)
	Sample ID number
	Date and time of collection
	Number and type of containers filled
	Analysis(es) requested
	Preservatives used (if applicable)
	Sample designation (grab or composite)
	Special instructions (for example, laboratory needs to sub-sample oversized material or perform additional homogenization)
	Signatures of all samplers
	Signatures of individuals involved in custody transfer, including the date and time of transfer
	Airbill number (if applicable)
	Project contact and telephone number

samples being shipped to a CLP Laboratory during collection, transportation, storage, and analysis in

Custody seal number

SulTRAC will follow the procedures in the EPA Region 5 CRL "Superfund Amendments and Reauthorization Act (SARA)/Superfund Sample Handling Manual" (EPA 1989) to complete the documentation listed above.

The XRF technician will also serve as the sample custodian. Upon completion of all required documents, the sample custodian will sign and date the documents and list the time of sample collection. The custodian also will confirm the completeness of all descriptive information on the COC forms, which will be included with each shipping container. Two custody seals will be used: one across the latch of the sample shipment container and the other on the opposite side of the container lid. The lid will be securely taped shut for shipment. The field sample custodian will send the original copies of the COC region copies to the project manager, who in turn will submit these to the Region 5 Sample Management Office (SMO) within 5 working days of the work completed. The sample custodian will also scan and retain copies of all COCs (laboratory and region) for the project files.

#### A8.6 SAMPLE PACKING AND SHIPMENT

The procedures to ship samples collected during this project to a CLP Laboratory are summarized below:

All sample jars will be individually wrapped with bubble wrap or other packing material and placed in their own individual ziplock-type bags. Each sample will have its CLP ID tag (if needed) accompanying the sample package.
Ice will be double-bagged in large ziplock-type bags and placed at the bottom of the shipping container. If the shipping container has a drain, the drain will be taped shut both inside and outside the shipping container.
The shipping container will be lined with bubble wrap or other packing material, and all individually packaged samples will be placed into one large plastic bag and tied shut. Sufficient packing material will be used to prevent sample containers from breaking during shipment.
Additional double-bagged ice will be added on top of the tied plastic bag full of samples. Enough ice will be added to maintain a sample temperature of $4 \pm 2$ °C. SulTRAC shall prepare, label, and place a temperature blank in each shipping container. SulTRAC shall also include one trip blank in each shipping container.
If a sampler suspects that any sample contains anomalously high or low concentrations or requires laboratory personnel to take safety precautions, this information will be handwritten directly on the laboratory copy of the COC form.
The COC form specific to each shipping container will be sealed inside a plastic bag and taped to the inside of the shipping container lid. The COC must be signed by all samplers and the custody seal numbers included on the COC form. A return prepaid airbill will be included with the COC form so the sample shipping container can be returned to SulTRAC.
The shipping container will be closed and taped shut with strapping tape around both ends.
Signed and dated custody seals will be placed on the front and side of each shipping container. Wide clear tape will be placed over the seals to prevent accidental tearing.

The airbill, if required, will be completed before the samples are relinquished to the carrier.
The COC form will be transported within the taped and sealed shipping container. When the shipping container is received at the analytical laboratory, laboratory personnel will open the shipping container and sign the COC form to document transfer of the samples.
The Superfund SMO will be notified if the laboratory expects to receive samples on Saturday. SulTRAC will call its CLP sample coordinator, who in turn will notify the SMO.

All shipping containers will be labeled as required by the U.S. Department of Transportation (DOT). After packing, the samples will be shipped to the CLP laboratory specified by the EPA Region 5 Regional Sample Control Coordinator.

#### A9.0 DISPOSAL OF INVESTIGATION-DERIVED WASTE

Investigation-derived waste (IDW) is expected to be minimal. Composite sample soil that is not submitted to a laboratory will be placed back into the augered holes at each property. Equipment will be decontaminated at each yard of each property, and rinsate will be discarded on the ground surface in the yard from which the samples were collected.

#### A10.0 HEALTH AND SAFETY PROCEDURES

Field activities will be conducted in accordance with the SulTRAC health and safety plan (HASP) (SulTRAC 2014b). Prior to initiation of field activities, SulTRAC field personnel and subcontractors will read and sign the HASP, indicating that they understand the HASP and agree to operate in accordance with its requirements. SulTRAC personnel and subcontractors must have 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) training, and proof of certification must be filed with the corporate health and Safety Officer and be available by request. At least one on-site person will have First Aid and cardiopulmonary resuscitation (CPR) training. A complete copy of the site-specific plans, including the updated HASP, will be maintained by the field sampling team at the site. Daily Safety Tailgate Meetings will be documented in the field log books and each employee will sign off on the Daily Tailgate Safety Meeting form as described in the HASP.

# A11.0 QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

All QA activities will be conducted in accordance with the SAP. A copy of the SAP will be maintained by the field sampling team for immediate use in resolving any QA issues that might arise during field activities.

#### A12.0 REFERENCES

- SulTRAC 2012a. "Remedial Investigation Report, US Smelter and Lead Refinery Superfund Site, Lake County, Indiana." June 20.
- SulTRAC 2012b. "Feasibility Study, US Smelter and Lead Refinery Superfund Site, Lake County, Indiana." June 20.
- SulTRAC 2014a. "Work Plan for USS Lead Superfund Site, East Chicago, Lake County, Indiana." August 6.
- SulTRAC 2014b. "Health and Safety Plan for USS Lead Superfund Site, East Chicago, Lake County, Indiana." December 12.
- SulTRAC 2014c. "Quality Assurance Project Plan for U.S. Smelter and Lead Residential Area Superfund Site, East Chicago, Lake County, Indiana". December 12.
- U.S. Environmental Protection Agency (U.S. EPA) 2000. "Technical Review Workgroup Recommendations for Sampling and Analysis of Soil at Lead (Pb) Sites." EPA-540-F-00-010, OSWER #9285.7-38. April. http://www.epa.gov/superfund/lead/products/sssiev.pdf
- U.S. EPA 2003. "Superfund Lead-Contaminated Residential Sites Handbook." August.
- U.S. EPA 2008. Hazardous Ranking System Documentation Record. US Smelter and Lead Refinery. April.
- U.S. EPA 2009. "Federal On-Scene Coordinator's Report, Rev. 1, Comprehensive Environmental Response, Compensation, and Liability Act Removal Action at the USS Lead Site, East Chicago, Lake County, Indiana, Site ID: 053J." August.
- U.S. EPA 2010. "EPA Contract Laboratory Program Statement of Work for Inorganic Superfund Methods, Multi-Media, Multi-Concentration, ISM01.3." January. On-line Address: http://www.epa.gov/superfund/programs/clp/ism1.htm
- U.S. EPA 2011. "Contract Laboratory Program Guidance for Field Samplers." January.
- U.S. EPA 2014. "RAC II Region 5 Statement of Work for Remedial Design (OU1), U.S. Smelter and Lead Residential Area Superfund Site." June 17.

FIGURES (Two Sheets)	
(Two Sheets)	
☐ FIGURE A-1, SITE LOCATION MAP☐ FIGURE A-2, USS LEAD RESIDENTIAL ARI	EA ZONES





# ATTACHMENT A

# STANDARD OPERATING PROCEDURES

SOP 001	Packaging, Shipping, and Custody of Samples, Revision No. 02. May 2013
SOP 002	General Equipment Decontamination, Revision No. 01. May 2013
SOP 003	Recording Notes In Field Logbook, Revision No. 01. May 2013
SOP 005	Soil Sampling, Revision No. 2, August 2013
SOP XRF	EPA Method 6200: Field X-Ray Fluorescence Measurement, Number: SESDPROC-
	107IR2, Revision 2, December 2011

# STANDARD OPERATING PROCEDURE (SOP) APPROVAL FORM

# SULLIVAN INTERNATIONAL GROUP, INC. ENVIRONMENTAL SOP

# PACKAGING, SHIPPING, AND CUSTODY OF SAMPLES SOP NO. 001 REVISION NO. 02

May 24, 2013

Merry Coons, P.E., QA/QC Approved 5/24/2013

Date

R5 USS LEAD FOIA 05/15/2017 ED\_000981\_00000421-00041

#### 1.0 PURPOSE AND SCOPE

Use of this Standard Operating Procedure (SOP) is critical to sample integrity. In this case, integrity is defined as the documentation and physical handling of a sample that allows the sample to be considered representative of an environmental medium at the time of collection. In a sampling program that meets the standard of care, the integrity of a sample is documented from its point and time of collection to its final disposition. Practices for classifying, packaging, and shipping samples are described in this SOP. Steps identified should be followed to ensure sample integrity and to protect the welfare of persons involved in shipping and receiving samples.

When hazardous substances and dangerous goods are sent by common carrier, their packaging, labeling, and shipping are regulated by the U.S. Department of Transportation (DOT) Hazardous Materials Regulations ([HMR] Code of Federal Regulations, Title 49 [49 CFR] Parts 105 through 180) and the International Air Transportation Association (IATA) Dangerous Goods Regulations (DGR), Section 1.5. This SOP only addresses materials shipping and transport that are not considered dangerous goods. Procedures for packaging and shipping environmental samples not considered dangerous goods are discussed below.

This SOP establishes the requirements and procedures for packaging and shipping samples. It has been prepared in accordance with the U.S. Environmental Protection Agency (EPA) "Sampler's Guide to the Contract Laboratory Program (CLP)," dated January 2011. There are small differences between the sample packaging for delivery to a CLP laboratory and for sample packaging for delivery to a commercial laboratory. This SOP notes where there are differences so that field staff will be informed of the proper procedures for the type of laboratory used. Sample packaging and shipping procedures described in this SOP should be followed for sample packaging and shipping. Deviations from the procedures in this SOP and the justification must be documented in a field logbook. This SOP assumes that samples are not dangerous goods and are already collected in the appropriate sample jars.

This SOP applies to sample custody, documentation, packaging, and shipping. The EPA regional Sample Management Office (SMO) is responsible for management, operations, and administrative support in the CLP.

This SOP applies to all Sullivan employees and employees of JV partner companies where Sullivan is the managing partner.

#### 2.0 PROCEDURES

The procedures for packaging and shipping samples that are not considered dangerous goods require the following:

- Coolers
- Ice

- Bubble wrap or equivalent cushioning material
- Chain-of-custody forms and seals
- Airbills
- Resealable plastic bags for sample jars and ice
- Packing tape
- Plastic trash bags to line the coolers prior to packing

The following procedures apply to packaging and shipping samples that are considered dangerous goods and samples that are not considered dangerous goods.

### 2.1 SAMPLE CUSTODY

Sample custody procedures are designed to ensure that sample integrity is maintained from collection to final disposition. A critical aspect of sound sample collection and analysis protocols is the maintenance of strict chain-of-custody procedures as described in this SOP. Chain-of-custody procedures include tracking and documentation during sample collection, shipment, and laboratory processing. A sample is considered to be in an individual's custody if it is (1) in the physical possession of the responsible party; (2) in view of the responsible party after being in their possession; (3) secured to prevent tampering; or (4) placed in a designated, secure area that is controlled and restricted by the responsible party.

Custody will be documented throughout all sampling activities on the chain-of-custody record for each sampling day. This record will accompany the samples from the site to the laboratory. All personnel who have custody of samples are required to sign, date, and note on the record the time when receiving and relinquishing samples from their immediate custody. Any discrepancies will be noted at this time. Samples will be shipped to subcontractor laboratories via overnight air courier or hand delivered. Any exceptions to this policy will be noted in the Project Sampling and Analysis Plan (SAP). Air Bills or bills of lading will be used as proof of custody during sample transport and will be retained as part of the permanent sample custody documentation. In some cases, samples may be hand delivered to the subcontractor laboratory. Hand delivery will be noted on the chain-of-custody (COC) form (Section 2.4). The subcontractor laboratory is responsible for sample custody after samples are received.

#### 2.2 SAMPLE CLASSIFICATION

Prior to sample shipment, it must be determined if samples collected during a specific field investigation meet the definitions for dangerous goods. This determination is made by an individual trained in dangerous goods shipping procedures. If a sample is collected of a material that is listed in the Dangerous Goods List, Section 4.2, IATA, then that sample must be identified, packaged, marked, labeled, and shipped according to the instructions given for that

material. If the composition of the collected sample(s) is unknown, and the project team knows or suspects that it is a regulated material (dangerous goods), the sample may not be submitted for air transport. If the composition and properties of a waste sample or highly contaminated soil, sediment, or water sample are unknown, or only partially known, the sample may not be offered for air transport. In addition, the shipment of pre-preserved sample containers or bottles of preservatives (e.g., NaOH pellets, HCL, etc.) which are designated as dangerous goods by IATA is regulated. Shipment of nitric acid is strictly regulated. Consult the IATA Dangerous Goods Regulations for guidance. Dangerous goods must not be offered for air transport by any personnel except the dangerous goods shipment designee or other personnel trained and certified by IATA in dangerous goods shipment. If coolers are to be used to ship dangerous goods, the environmental samples should be a limited quantity of no more than 30 kg (66 pounds) per shipment. If the weight of the sample material is greater than 30 kg per shipment, then dangerous goods shipping standards must be followed.

### 2.3 PACKAGING ENVIRONMENTAL SAMPLES

Environmental samples, after being correctly containerized, should be labeled. If the samples are to be analyzed by a commercial (non-CLP) laboratory, then the commercial laboratory will provide sample container labels. If no other label is available, the Sample Label may be used (included in the Forms section of this SOP). The following information is required to be written on the label:

- Sample ID Sample nomenclature should be taken from the Project Sampling and Analysis Plan (SAP).
- Sample Date The date the specific sample was collected.
- Sample Time The time the specific sample was collected.
- Location The location of the sample collected. For example, this information may be a monitoring well identification or a soil boring identification.
- Preservative If a preservative, such as hydrochloric acid, is used, the information should be included on the label to let the laboratory staff be prepared to safely handle the sample during analysis.
- Analysis List the analysis(es) required for the sample collected.
- Name or initials of the person(s) collecting the sample.

The label for each CLP sample should contain the following information:

Case Number – This is the number provided by the SMO that is site and sampling
event specific. It must be on all samples so the CLP laboratory knows to which
Superfund project it belongs.

- Station Location This is usually the sample location, such as monitoring well MW-XXX or surface soil location SS-XXX.
- Sample Number This is the CLP sample identification number provided by the regional SMO. It is unique to a sample location and is used to identify and track samples throughout the sampling and analytical processes.
- Sample Date The date the specific sample was collected.
- Sample Time The time the specific sample was collected.
- Analysis List the analysis(es) required for the sample collected.
- Tag Number –This is the seven to eight character number on the pre-printed tags supplied by the Regional SMO.

Prior to submitting the samples to the laboratory, the following steps should be followed to properly package the samples. Additional steps for proper packaging of CLP samples are noted below.

- Seal all drain holes in the cooler, if any, to prevent any leaking if samples break or from ice melt.
- Place completed label on the sample container. Note: Soil samples collected for volatile organic analyses (VOAs) using a 40-ml glass vial should not have the printed labels attached to the 40-ml vials. These sample vials already have a label attached. The attached label will have a tare weight on it. This means that the vial with the label has been weighed on a laboratory grade scale. It is important that nothing except the soil sample is in, or on, the vial. Information from the label prepared in the field can be written on the already attached vial label. Insert the field printed label in the resealable plastic bag discussed in item 6 below.
- Use clear packing tape to cover the label. This protects it from moisture that might cause it to detach from the container.
- For CLP samples, attach the Sample Tag to the container. Information on the sample label should match the information on the Sample Tag.
- If the sample container is glass, wrap the labeled sample in bubble wrap.
- Place the sample in a re-sealable plastic bag; no more than one container per bag is recommended, but you can wrap 40 ml vials in bubble wrap and place two or three to a bag.

- Place the bagged sample in a cooler. It is recommended that the cooler be lined with a large plastic garbage bag before anything else is placed in the cooler to contain sample material in case on container breakage.
- If additional cushioning of the samples is required, use bubble wrap to keep the glass containers from breaking. Do not use vermiculite or cat litter as sources of packing material.
- Add a sufficient quantity of ice to the cooler to cool samples to 4 °C. Ice should be double bagged in re-sealable plastic gallon-sized bags to prevent the melted ice from leaking out. It is recommended that two gallon-sized bags of ice be placed on the bottom of the cooler beneath the samples and two gallon-sized bags of ice be placed on top of the packaged samples.
- A temperature blank (a sample bottle filled with tap water) should be included with the cooler.
- Seal the completed, and signed, chain-of-custody forms in a plastic bag, after removing the back copy, and tape the plastic bag to the inside of the cooler lid.
- If the cooler is to be returned, include instructions for returning the cooler with the chain-of-custody forms.
- Close the lid of the cooler and tape it shut by wrapping packing tape around both ends and hinges of the cooler multiple times.. (Note: Sampling is expensive and tape is inexpensive, so use much more tape than you think is necessary to keep the cooler securely closed during transport.)
- Place two custody seals on the cooler, ensuring that one covers the cooler lid and the other on a hinge on the back side of the cooler. Place clear plastic tape over the custody seals. When Laboratory couriers are used to pick up coolers from the jobsite, this step is not applicable. If no other seal is available, the Sample Seal may be used (included in the Forms section of this SOP).
- Place address labels on the outside of the cooler. When Laboratory couriers are used to pick up coolers from the jobsite, this step is not applicable.
- Ship samples overnight by a commercial carrier such as FedEx. When Laboratory couriers are used to pick up coolers from the jobsite, this step is not applicable.

## 2.4 TRAFFIC REPORTS/CHAIN-OF-CUSTODY FORMS FOR SHIPPING SAMPLES

The traffic report (TR), or COC form, is used as physical evidence of sample custody from the sample location to the analytical laboratory. It is a permanent record for each sample collected and transported.

For commercial laboratories, the laboratory will provide COC forms along with labels and containers as specified in their contract with Sullivan. If no other COC form is available, the Chain-of-Custody Record form may be used (included in the Forms section of this SOP). The COC form is completed by the sampler or designated sample custodian. If the laboratory provides pick up service, the laboratory courier signs the COC form and the Sullivan sampler retains a copy for the project records. If the samples are shipped by an express commercial carrier, such as FedEx, the COC form is enclosed in the cooler as discussed in Section 2.2 above.

For CLP laboratories, Scribe software is used to create the documentation, including the TR/COC form, for the samples. Samples should be grouped by laboratory receiving them. When the sampling event Case Number is assigned, laboratory assignments will be divided between inorganic analysis and organic analysis depending on the types of chemical analysis requested.

For CLP sample shipments to the laboratory(s), a TR must be completed for each sample cooler. Each sample cooler will contain the TR/COC form specific to the samples within the cooler. The TR will contain the following information:

- Date the samples are shipped.
- The carrier and carrier airbill number. Each cooler must have its own airbill multiple coolers should not be sent under one airbill.
- The laboratory and the laboratory contact information to which the samples are shipped.
- The Case Number assigned to the sampling event must be on every TR/COC form. The laboratory will not know to which Case a particular sample shipment is assigned unless it is on the TR/COC form.
- Information on the sample labels and tags should be included on the TR/COC form including the organic or inorganic CLP sample identification number, analysis, station location, tag number, and the date and time sample was collected.
- In addition, the following information should be included on the TR/COC form:
  - matrix type and sampler's last name;
  - the collection method, e.g., grab, composite, etc.;
  - turnaround time;
  - preservative;
  - number of containers per CLP sample identification number; and
  - the corresponding inorganic or organic sample identification number depending on what the TR/COC form is transmitting, e.g., if you are shipping organic samples, then the corresponding inorganic sample identification number.

The Scribe software program automatically generates a Traffic Report number which should be included on the TR/COC form. If any samples are to be analyzed using a CLP Modified Analysis (MA), the sampler should indicate use of the MA by creating a new analysis within the Scribe Analysis table. The newly created analysis should contain the Modification Reference Number within the name assigned in the analysis.

## 2.5 OVERNIGHT OR INDEFINITE SAMPLE STORAGE

In some cases, samples that cannot be shipped immediately to a laboratory must be temporarily stored in a secure location or sample refrigerator/cooler until arrangements can be made for delivery. The Sample Custodian shall place samples in the designated location (samples and signed chain of custody record(s) in re-sealable bags) and secure the samples until their disposition is determined. A temperature blank must accompany samples stored for any duration that require cooling as a preservation technique.

#### **GLOSSARY**

**Contract Laboratory Program:** CLP is the USEPA's Superfund laboratory program that provides analytical services through a national system which is required to provide data of known and documented quality.

Custody seal: A custody seal documents that no tampering with the samples occurred after they have been packaged for shipping. A CLP custody seal is a tape-like seal pre-printed with a specific number provided by the USEPA Region of origin. A non-CLP custody seal is a tape-like seal either pre-printed with the commercial laboratory's information and/or with the sampler's initials and the date. The custody seal is placed across the cooler lid and considered part of the chain-of-custody process.

**Dangerous goods:** Dangerous goods are articles or substances that can pose a risk to health, safety, property, or the environment and which are shown in the list of dangerous goods or which are classified according to the regulations (Section 1.0) based on the latest IATA DGR manual.

**Environmental samples:** Environmental samples are specimens collected for laboratory analysis. Sampled media may include, but are not limited to, drinking water, groundwater and ambient surface water, soil, sediment, air, and biological (e.g., tissue or plant) specimens.

**Hazardous Materials Regulations:** The HMR are DOT regulations for the shipment of hazardous materials by air, water, and land; they are located in 49 CFR 105 through 180.

**Hazardous substance:** A hazardous substance is any material, including its mixtures and solutions, that is listed in Appendix A of 49 CFR 172.101 and its quantity, in one package, equals or exceeds the reportable quantity (RQ) listed in the appendix.

**IATA Dangerous Goods Regulations:** The DGR are regulations that govern the international transport of dangerous goods by air. The IATA DGR are updated annually and posted in September. The DGR are based on the International Civil Aviation Organization (ICAO) Technical Instructions (49 CFR 171.11). The DGR contain all of the requirements of the ICAO Technical Instructions and are more restrictive in some instances.

**Modified Analysis:** The Modified Analysis (MA) is an analytical method that is not considered routine under the CLP. This is established during the analytical services request process. A modified analysis to be performed by a specific analytical laboratory will receive a unique MA number. For example, if the Region submits Modified Analysis for an additional analyte, the laboratory assignment will include the Modification Reference Number, such as VOC by MA 1100.

**Overpack:** An enclosure used by a single shipper to contain one or more packages and to form one handling unit for convenience of handling and stowage. Dangerous goods packages contained in the overpack must be properly packed, marked, labeled and in proper condition as required by IATA regulations (the latest edition of the IATA DGR). For example, a cardboard box may be used to contain three fiberboard boxes to make handling easier and to save on shipping costs if the cardboard box meets the requirement for an outer box.

**Sample Label:** A record attached to sample containers to verify legal documentation of traceability.

Title: Packaging, Shipping, and Custody of Samples

#### REFERENCES

- U.S. Department of Transportation, Transport Canada, and the Secretariat of Communications and Transportation of Mexico (DOT and others). 2008. "2008 North American Emergency Response Guidebook."
- International Air Transport Association (IATA). 2011. "Guidelines for Instructors of Dangerous Courses."
- IATA. 2011. "Dangerous Goods Regulations." 52nd Edition.
- U.S. Environmental Protection Agency. 2011. "Sampler's Guide to the Contract Laboratory Program." Office of Solid Waste and Emergency Response. Washington, D.C. EPA/540/R-96/032. On-Line Address:
  - http://www.epa.gov/oerrpage/superfund/programs/clp/guidance.htm#sample

**FORMS** 

# Sample Label

Sample Information	
Sample ID	Sample Date
Location	Sample Time
Analysis	Preservative
Site	Sampled By

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# STANDARD OPERATING PROCEDURE (SOP) APPROVAL FORM

# SULLIVAN INTERNATIONAL GROUP, INC. ENVIRONMENTAL SOP

# GENERAL EQUIPMENT DECONTAMINATION SOP NO. 002 REVISION NO. 01

May 24, 2013

	5/24/2013
Richard Baldino, Author	Date
	5/24/2013
Tessa McRae, Technical Review	Date
	5/24/2013
Merry Coons, P.F. QA/QC Approved	Date

R5 USS LEAD FOIA 05/15/2017 ED\_000981\_00000421-00056

#### 1.0 PURPOSE AND SCOPE

The purpose of this standard operating procedure (SOP) is to provide methods to be used for preventing, minimizing, or limiting cross-contamination of samples as well as for reducing or eliminating the transfer of contaminants to clean areas. All non-disposable field equipment must be decontaminated before and after each use at each sampling location to obtain representative samples. This SOP is applicable to standard field investigation activities (i.e., soil, groundwater, surface water, and sediment sampling) and does not address personnel decontamination. Decontamination of specialized materials such as radioactive materials is not addressed in this SOP.

Decontamination method(s) can be selected based on site-specific conditions; therefore, this SOP is not intended to be used as a decision document. Rather, its intended use is as a supplement to the work planning process by providing procedures, current references, and options for decontamination of equipment.

The primary reference for this SOP is The EPA "Sampling Equipment Decontamination" Environmental Response Team SOP #2006 (Rev. #0.0, 08/11/94).

This SOP applies to all Sullivan employees and employees of JV partner companies where Sullivan is the managing partner.

## 2.0 METHODS AND PROCEDURES

Gross contamination can be removed by physical decontamination procedures such as brushing, air and wet blasting, and high- and low-pressure water cleaning. Depending on the chemicals present, an acid rinse and/or a high-purity organic solvent rinse may be required to remove the chemical(s) of concern. The following general steps must be taken when decontaminating sampling materials and equipment:

- 1. Physical removal by brushing, air and/or wet blasting, and high- or low-pressure water cleaning.
- 2. Non-phosphate detergent wash.
- 3. Tap-water rinse.
- 4. Distilled/deionized water rinse.
- \*\*The following steps are optional; one or more may be specified in the project-specific Sampling and Analysis Plan (SAP)\*\*
- 5. 10% nitric acid rinse (for decontamination of metals).
- 6. Distilled/deionized water rinse.
- 7. Solvent rinse (acetone is commonly used to remove trace organic contaminants).
- 8. Air dry (if solvent rinse is used).
- 9. Distilled/deionized water rinse (if solvent rinse is used).

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Depending upon the type and size of the equipment needing decontamination, large galvanized wash tubs, stock tanks, children's wading pool, or buckets can hold wash and rinse solutions. All decontamination water generated must be containerized and disposed of according to Sullivan Environmental SOP No. 16, Investigation-Derived Waste Management, or the site-specific project plan.

The following procedures present specific information and procedures for decontaminating drilling and monitoring well installation equipment, borehole soil sampling equipment, water level measurement equipment, and general sampling equipment.

# 2.1 DECONTAMINATION OF DRILLING AND MONITORING WELL INSTALLATION EQUIPMENT

All drilling equipment that is reused during the sampling process, such as drilling rods, must be decontaminated at a designated location onsite (if possible) before drilling operations begin, between borings, and at completion of the project. All permanent monitoring well casings, screens, and fittings are assumed to be delivered to the site in a clean condition, which must be verified by the field personnel. If a temporary stainless-steel well screen is used to collect grab groundwater samples by direct-push technology, the well screen must be decontaminated after each groundwater sample collection.

Various methods will remove the contaminants that adhere to the drilling equipment. Typically, the drilling subcontractor will use a physical approach to decontaminate the equipment. The following steps must be taken when decontaminating drilling equipment:

- 1. Pressure-wash the down-hole equipment with water to remove large particles of soil.
- 2. Wash equipment with a scrub brush or wire brush in a tap water or distilled water and non-phosphate detergent solution such as Liquinox® or Alconox®.
- 3. Rinse the equipment with distilled water and pressure-wash a second time. Make sure that decontaminated rods are not placed together with contaminated rods.

#### 2.2 DECONTAMINATION OF BOREHOLE SOIL-SAMPLING EQUIPMENT

The soil sampling equipment must be decontaminated after each sample as follows:

- 1. Prior to sampling, scrub the split-barrel sampler and sampling tools in a bucket using a stiff, long-handled scrub brush and Liquinox® or Alconox® solution.
- 2. Steam clean the sampling equipment over the rinsate tub and allow to air dry.
- 3. Place cleaned equipment in a clean area on plastic sheeting.
- 4. Containerize all wastewater.
- 5. Decontaminate all pipe placed down the hole as described for drilling equipment.

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## 2.3 DECONTAMINATION OF WATER LEVEL MEASUREMENT EQUIPMENT

Field personnel must decontaminate the water level indicator sounder and interface probe before inserting and after removing them from each well. The following decontamination procedures must be used:

- 1. Wipe the sounding cable and probe with a disposable soap-impregnated cloth or paper towel.
- 2. Rinse with distilled/deionized water.

#### 2.4 DECONTAMINATION OF GROUNDWATER PUMPS

Field personnel must decontaminate the submersible pumps before inserting and after removing them from each well. All submersible water pumps used for groundwater sampling or well development must be decontaminated using the following procedures:

- 1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
- 2. Maintain the same level of personal protective equipment (PPE) as was used for sampling.
- 3. To decontaminate a submersible pump, all of the following steps shall be followed as in addition to what is specified in the project-specific Field Sampling Plan (FSP):
  - a. Fill a clean bucket with tap water and sulfate-free detergent (i.e., Liquinox® or Alconox®). The bucket must be large enough to immerse the pump inlets in water.
  - b. Pump the tap water with sulfate-free detergent through the equipment.
  - c. Repeat with a second bucket containing only tap water.
  - d Repeat with a third bucket containing only distilled/deionized water.

Note: Do **not** use regular commercial grade soap in the pumps.

#### 2.5 DECONTAMINATION OF WATER QUALITY METERS

Field personnel must decontaminate the flow-through cells and the water-quality probes before and after using them at each well. The flow-through cells and associated probes must be decontaminated using the following procedures:

- 1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
- 2. Maintain the same level of PPE as was used for sampling.

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- 3. To decontaminate the flow-through cell, all of the following steps shall be followed in addition to what is specified in the project-specific FSP:
  - a. Liquinox® or Alconox® wash
  - b. Tap water rinse
  - c. Distilled/deionized water rinse
- 4. To decontaminate the water-quality probes that fit in the flow-through cell, all of the following steps shall be followed as specified in the project-specific FSP:
  - a. Liquinox® or Alconox® wash
  - b. Tap water rinse
  - c. Distilled/deionized water rinse

Note: Do not use regular commercial grade soap in the cells or probes.

# 2.6 DECONTAMINATION OF GENERAL SAMPLING EQUIPMENT

All non-disposable sampling equipment must be decontaminated using the following procedures:

- 1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
- 2. Maintain the same level of PPE as was used for sampling.
- 3. To decontaminate a piece of equipment, some or all of the following steps shall be followed in addition to what is specified in the project-specific FSP:
  - a. Liquinox® or Alconox® wash
  - b. Tap water rinse
  - c. Distilled/deionized water rinse
  - \*\*Optional steps as specified in the project specific SAP\*\*
  - d. 10 percent nitric acid rinse (used only if samples are collected for metals analysis)
  - e. Distilled/deionized water rinse
  - f. Solvent rinse (HPLC grade acetone, hexane, or methanol; used only if samples are collected for organic analysis)
  - g. Allow the solvent to evaporate
  - h. Distilled/deionized water rinse
  - i. Place cleaned equipment in a clean area on plastic sheeting, allow to air dry, and wrap with aluminum foil
- 4. Containerize all wastewater.
- 5. Containerize all PPE generated during decontamination efforts as described in the site-specific project plan and Sullivan Environmental SOP No. 16, Investigation-Derived Waste Management.

Title: General Equipment Decontamination

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## **GLOSSARY**

**Split-Spoon (or Split-Barrel) Sampler:** Thick-walled steel tube that is split lengthwise. A cutting shoe is attached to the lower end; the upper end contains a check valve and is connected to drill rods.

**Thin-Wall Tube Sampler:** Steel tube (1 to 3 millimeters thick) with tapered bottom edge for cutting. The upper end is fastened to a check valve that is attached to drill rods. A plastic catcher may be included inside the cutting shoe when sampling sandy soils to reduce sample loss.

Title: General Equipment Decontamination

Page 6 of 6 Revision No. 01 Date: May 24, 2013

# **REFERENCES**

U.S. Environmental Protection Agency (EPA). 1994. "Sampling Equipment Decontamination." Environmental Response Team SOP #2006, Rev # 0.0. August 11.

# STANDARD OPERATING PROCEDURE (SOP) APPROVAL FORM

# SULLIVAN INTERNATIONAL GROUP, INC. ENVIRONMENTAL SOP

# RECORDING NOTES IN FIELD LOGBOOK SOP NO. 003 REVISION NO. 01

May 24, 2013

Richard Baldino, Author	<u>5/24/2013</u> Date
Tessa McRae, Technical Review	<u>5/24/2013</u> Date
Merry Coons, P.E., QA/QC Approved	<u>5/24/2013</u> Date

R5 USS LEAD FOIA 05/15/2017

Title: Recording Notes in Field Logbook

Page 1 of 6 Revision No. 01 Date: May 24, 2013

#### 1.0 PURPOSE AND SCOPE

During field activity, personnel perform the tasks described in the work plan or contract. In addition to performing the field tasks, personnel are required to maintain detailed records of the field activities, observations, and conditions encountered at the site. Entries must be described in sufficient detail so that activities and events can be reconstructed in the future.

Although there are many mechanisms for recording data, the purpose of this standard operating procedure (SOP) is to provide clear guidance to verify that logbook documentation is correct, and adequate. This SOP establishes the general requirements and procedures for recording notes in the field logbook. The primary reference for this SOP is *A Manual of Field Hydrogeology*, Laura L. Sanders, Northeastern Illinois University; Prentice Hall (1998). However, many of the practices described in this SOP are based in legal and/or accepted industry practices.

Logbooks are considered official documents that can be used during enforcement proceedings and may be subject to legal or professional review at any time. Therefore, the entries in the logbook must be accurate, detailed, and reflect the importance of the field events.

Logbooks are used for a variety of purposes, but primarily are used to document: sampling activity; activity relating to, and deviations from, the project approach, work plans, quality assurance project plans, and health and safety plans; project personnel; photographic information, weather, keen observations of site conditions, and interaction with the public or media. Data recorded in the logbook assist in accurate figure generation and analytical result interpretation. A complete and accurate logbook enhances quality control by proper documentation of field observations, activities, and decisions.

This SOP applies to all Sullivan employees and employees of JV partner companies where Sullivan is the managing partner.

## 2.0 REQUIREMENTS AND RESOURCES

The following items are required for field notation:

Field logbook
Ballpoint pens with permanent ink
 6-inch ruler (optional)

Field logbooks are available in a variety of formats. Logbooks must be bound, water-resistant, and must contain pre-printed lines and page numbers. Loose-leaf paper is not an acceptable format; however, if notes are recorded on loose-leaf paper, the notes must be transcribed into a logbook as soon as possible by the original note recorder. Pre-printed forms (water-quality data sheets, soil boring logs, etc.) must be kept in waterproof containers such as plastic sleeves or clipboards with a cover.

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Logbooks can typically be obtained through a document control administrator (DCA), project manager, or field team leader and are available from geological, forestry, or surveying supply houses. In some instances, logbooks may have a designation or specific identification number.

## 3.0 PROCEDURES

The following sub-sections present general guidelines and formatting requirements for field logbooks and detailed procedures for completing field logbooks.

## 3.1 REQUIREMENTS AND GENERAL GUIDELINES

	A separate field logbook must be maintained for each project. If a site consists of multiple sub-sites, designate a separate logbook for each sub-site. For special tasks, such as periodic well-water-level measurements, data from multiple sub-sites may be entered into one logbook which contains only one type of information.
	If a client requires separate, project-specific field sheets in addition to the logbook, then this fact must be noted in the logbook together with a notation specifying where the completed sheets can be found.
	As stated above, logbooks must be bound and contain consecutively numbered pages.
	Pages may not be removed from the logbook for any purpose.
enanting (	Logbooks pertaining to a site or sub-site must be assigned a consecutive serial number.
	The project manager must maintain a record of all logbooks issued under the project.
	Information must be entered with a ballpoint pen with waterproof ink. To avoid smearing, do not use pens with "wet ink." The ink must be clearly visible and easily photocopied.
( common	Pencils are generally not permissible for field notes because information can be erased. However, in extremely cold conditions where pen use is not possible, pencils may be used. The reason for pencil entries must be clearly stated in the logbook.
	Irrelevant information must not be entered in the logbook. Language used in the logbook must be factual and objective.
	Record observations while in the field at the time the observation is made. Entries made at a later time or date must be noted as such.
	Begin a new page at the beginning of each day.

		ernanonal Group, Inc. – Environmental SOP No. 003  eding Notes in Field Logbook	Revision No. 01 Date: May 24, 2013
		If a subject changes and an additional blank space is necessary to nesubject title stand out, skip one line before beginning the new subject any pages or parts of pages unless a day's activity ends in the midd	ct. Do not skip
		Draw a diagonal line on any blank spaces of four lines or more to punauthorized entries.	revent
		Leave a wide margin and line spaces between entries to keep the lo organized. Record changes in weather and site conditions in the spathe margins.	•
		Use a tabulated format if possible within the logbook when recording information, particularly numerical data.	ng repetitive
3.2		LOGBOOK FORMAT	
	•	at and organization of each field logbook must be consistent with otes for the cover, spine, and internal pagination are discussed below.	her field logbooks.
3.2.1		Cover Designation	
Write	e the	following information in clear capital letters on the front cover of each	ch logbook.
		Logbook identification number (assigned by the DCA)	
		The serial number of the logbook (assigned by the project manager	)
		Name of the site, city, and state	
		Name of sub-site (if applicable)	
		Type of activity	
	***************************************	Beginning and ending dates of activities entered into the logbook	

Some of the information listed above, such as the list of activities and ending dates, must be entered after the entire logbook has been filled or after it has been decided that the remaining blank pages in the logbook will not be filled.

"Sullivan International Group, Inc." city and state

"REWARD IF FOUND"

The spine of the logbook must contain an abbreviated version of the information on the cover. For example: "Logbook 001, Acme Foundry, Des Moines, Iowa, April 2010 to May 2011."

Page 4 of 6 Revision No. 01 Date: May 24, 2013

# 3.2.2 Mailing Address and Contact Information

The inside front cover of most logbooks contains a lined field for entering the company name, address, and other contact information (telephone or mobile phone numbers, email addresses or websites). If pre-printed spaces for this information are not provided in the logbook, write the information on the first available page. Include the project manager's name and repeat "REWARD IF FOUND" under the entries.

## 3.3 LOGBOOK ENTRIES AND REQUIRED INFORMATION

Enter the following information at the beginning of each day or whenever warranted during the course of a day:

	Date
	Starting time
	Specific location
	General weather conditions and approximate temperature
	Names of personnel present at the site. Note the affiliation(s) and designation(s) of all personnel.
	Equipment calibration and equipment models used.
	Changes in instructions or activities at the site.
	Levels of personal protective clothing and equipment.
	A general title of the task undertaken (for example, well installation at MW-11, deconat borehole BH-11, groundwater sampling at MW-11).
Tanana Tanan	Provide an approximate scale for all diagrams. If this cannot be done, write "not to scale" on the diagram. Indicate the north direction on all maps and cross-sections. Label features on each diagram.
	Corrections must be made by drawing a single line through the entry being corrected. Initial and date any corrections made in the logbook.
	The person recording notes shall initial each page after the last entry. No information will be entered in the area following these initials.
	At the end of the day, the person recording notes shall sign and date the bottom of the last page. Indicate the end of the work day by writing "Left site at (time)." A diagonal line will be drawn across any blank space to the bottom of the page.

The follow	ving information must be recorded in the logbook after taking a photograph:
	Time, date, location, direction, and (if appropriate) weather conditions
	Description of the subject photographed and the reason for taking the picture
	Sequential number of the photograph and the film roll number, or the file location on the network (if applicable, i.e. a digital photograph saved on the Sullivan network)
	Name of the photographer
The follow	ving information must be entered into the logbook when collecting samples:
	Location description
	Names of samplers
	Collection time
	Designation of samples as a grab or composite sample
	Type of sample (water, sediment, soil gas, etc.)
	On-site measurement data (pH, temperature, specific conductivity)
	Field observations (odors, colors, weather, etc.)
	Preliminary sample description
	Requested analyses and preservatives
	Type of preservatives used
	Instrument readings

#### 3.4 CUSTODY

Custody of field logbooks must be maintained at all times. Field personnel must secure the logbook in a locked place (car, field office) when the logbook is not in their personal possession. When logbooks are not actively being used on a project site, it is the responsibility of the project manager to secure field logbooks in the project file or other similar project location that is both secure and accessible to project personnel.

Title: Recording Notes in Field Logbook

Page 6 of 6 Revision No. 01 Date: May 24, 2013

# **REFERENCES**

Compton, R.R. 1985. Geology in the Field. John Wiley and Sons. New York, N.Y.

Sanders, Laura L. 1998. *A Manual of Field Hydrogeology*. Northeastern Illinois University. Prentice Hall. March.

## **SOP APPROVAL FORM**



## PROJECT-SPECIFIC

## ENVIRONMENTAL STANDARD OPERATING PROCEDURE

**SOIL SAMPLING** 

**SOP NO. 005** 

**REVISION NO. 2** 

Last Reviewed: August 2013

Quality Assurance Approved

19 August 2013

Date

R5 USS LEAD FOIA 05/15/2017 ED\_000981\_00000421-00070

Last Reviewed: August 2013

#### 1.0 BACKGROUND

Soil sampling is conducted for three main reasons: for laboratory chemical analysis, laboratory physical analysis, or visual classification and field screening. These three sampling objectives can be achieved separately or in combination with each other. Sampling locations are typically chosen to provide chemical, physical, or visual information in both the horizontal and vertical directions. A sampling and analysis plan is used to outline sampling methods and provide preliminary rationale for sampling locations. Sampling locations may be adjusted in the field based on the screening methods being used and the physical features of the area.

#### 1.1 PURPOSE

Soil sampling is conducted to determine the chemical, physical, and visual characteristics of surface and subsurface soils.

#### 1.2 SCOPE

This standard operating procedure (SOP) describes procedures for soil sampling in different areas using various implements. It includes procedures for test pit, surface soil, and subsurface soil sampling, and describes ten soil sampling devices.

#### 1.3 **DEFINITIONS**

**Hand auger:** Instrument attached to the bottom of a length of pipe that has a crossarm or "T" handle at the top. The auger can be closed-spiral or open-spiral.

**Bucket auger:** A type of auger that consists of a cylindrical bucket 10 to 72 inches in diameter with teeth arranged at the bottom.

**Core sampler:** Thin-wall cylindrical metal tube with diameter of 0.5 to 3 inches, a tapered nosepiece, a "T" handle to facilitate sampler deployment and retrieval, and a check valve (flutter valve) in the headpiece.

EnCore<sup>TM</sup> sampler: A disposable volumetric sampling device. It comes in sample sizes of 5 and 25 grams. It is a hermetically sealed, single-use soil sampler made from a high-tech, inert polymer. EnCore<sup>TM</sup> samplers are used to collect soil samples with zero headspace, as required for volatile organic compound analysis. Each sample is collected using a reuseable "T" handle.

**Spatulas or Spoons:** Stainless steel or disposable instruments for collecting loose unconsolidated material.

**Trier:** Tube cut in half lengthwise with a sharpened tip that allows for collection of sticky solids or loosening of cohesive soils.

**Trowel:** Metal or disposable tool with a scooped blade 4 to 8 inches long and 2 to 3 inches wide with a handle.

**Split-Spoon (or Split-Barrel) Sampler:** Thick-walled steel tube that is split lengthwise. A cutting shoe is attached to the lower end; the upper end contains a check valve and is connected to drill rods.

**Thin-Wall Tube Sampler:** Steel tube (1 to 3 millimeters thick) with a tapered bottom edge for cutting. The upper end is fastened to a check valve that is attached to drill rods.

**Volatile Organics Analysis (VOA) Plunger:** Disposable, plastic, single-use soil sample collection device for volatile organic compound sample collection.

#### 1.4 REFERENCES

- U.S. Environmental Protection Agency. (EPA) 1984. "Soil Sampling Quality Assurance Users Guide." EPA 600/4-84-043.
- EPA. 1980. "Samplers and Sampling Procedures for Hazardous Waste Streams." EPA 600/2-80-018. January.
- EPA 1983. "Preparation of Soil Sampling Protocol: Techniques and Strategies." EPA 600/4-83-020.
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- EPA. 1991. "Handbook of Suggested Practices for the Design and Installation of Ground-Water Monitoring Wells." March. EPA/600/4-89/034.

EPA.	1994. "Soil Sampling." Environmental Response Team SOP #2012 (Rev. #0.0, 11/16/94) http://www.ert.org/products/2012.pdf
EPA.	1996. SW-846, Method 5035, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples. December. <a href="http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/5035.pdf">http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/5035.pdf</a>
1.5	REQUIREMENTS AND RESOURCES
Soil s	ampling requires the use of one or more of the following types of equipment:
	Spoons and spatulas
	Trowel
	Shovel or spade
	Trier
	Core sampler
	EnCore <sup>TM</sup> sampler
	VOA Plunger
	Hand auger
	Bucket auger
	Split-spoon
	Thin-wall tube
In add	lition, the following equipment is also needed for various methods:
	Sample containers, labels, and chain-of-custody forms
	Logbook
	Tape for measuring recovery
	Soil classification information
	Wax or caps for sealing ends of thin-wall tube
	"T" Handles
	Plastic sheeting
	Decontamination equipment
	Drilling equipment
	Backhoe
	Health and safety equipment

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#### 2.0 SOIL SAMPLING PROCEDURES

This SOP presents procedures for conducting test pit, surface soil, and subsurface soil sampling. The site sampling plan will specify which of the following procedures will be used.

Soil samples for chemical analysis should be collected in the following order: (1) volatile organics, (2) semivolatile organics, and (3) metals. Once the chemical samples have been containerized, samples for physical analyses can be containerized. Typical physical analyses conducted include (1) grain size distribution, (2) moisture content, (3) saturated permeability, (4) unsaturated permeability, and (5) Atterberg limits. Additionally, visual descriptions of samples, using the Unified Soil Classification System (USCS), should be recorded. Field tests such as head space analyses can also be conducted.

Soil samples for chemical analyses can be collected either as grab samples or composite samples. A grab sample is collected from a discrete location or depth. A composite sample consists of soil combined from more than one discrete location. Typically, composite samples consist of soil obtained from several locations and homogenized in a stainless steel or Teflon® pan, tray, or baggie. Refer to the site-specific Quality Assurance Project Plan (QAPP) for methodology for composite sample collection. Samples for volatile organics analysis should not be composited.

All soil samples collected should be packaged and shipped to the laboratories in accordance with SOP 019. All nondedicated or nondisposable equipment used for soil sampling should be decontaminated between sampling locations in accordance with SOP 002.

#### 2.1 SOIL SAMPLE COLLECTION PROCEDURES

Soil samples can be collected as discrete samples for volatile organic compound (VOC) analysis using specialized equipment for preservation in the laboratory or in the field. Soil samples collected for non-VOC analysis can be collected as either grab or composite samples using standard equipment.

#### 2.1.1 Procedure for Preserving and Collecting Soil Samples for VOC analysis

Samples collected for VOC analysis using traditional methods, such as collection in a jar with no preservation, are shown to yield nonrepresentative samples due to loss of VOCs. To prevent such losses,

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preservation with methanol or sodium bisulfite may be used to minimize volatilization and biodegradation. This preservation may be performed in the laboratory or in the field, depending on the sample collection methodology used. The specific sampling methodology will be specified in the project-specific QAPP or work plan.

Soil samples to be preserved in the laboratory are collected using SW-846 Method 5035. For samples preserved in the field, laboratories may perform low-level analysis (sodium bisulfate preservation) or high- to medium-level analyses (methanol preservation), depending on the project-specific QAPP.

The following procedures outline the necessary steps for collecting soil samples to be preserved at the laboratory, and for collecting soil samples to be preserved in the field with methanol or sodium bisulfate.

#### 2.1.1.1 Soil Samples to be Preserved at the Laboratory

Soil samples collected for VOC analysis that are to be preserved at the laboratory shall be obtained using a hand-operated, hermetically sealed sample vial such as an EnCore<sup>TM</sup> sampler. Each sample shall be obtained using a reusable sampling handle ("T" handle) that can be provided with the EnCore<sup>TM</sup> sampler when requested and purchased. Collect the soil sample in the following manner for each EnCore<sup>TM</sup> sampler.

The EnCore<sup>TM</sup> sampler is loaded into the "T" handle with the plunger fully depressed. Press the "T" handle into the soil to be sampled. The plunger will be forced upward as the cavity fills with soil. When the sampler is full, using the "T" handle, rotate the plunger and lock it into place. If the plunger does not lock, then it is not filled with soil. Soft soil may require several plunges or forcing soil against a hard surface such as a decontaminated sample trowel to ensure headspace has been eliminated. Remove soil from the outside of the sampler so a tight seal can be made between the sample cap and the O-ring. With soil slightly piled above the rim of the sampler, force the cap on until the catches hook the side of the sampler. Remove any surface soil from outside of the sampler and place in the foil bag provided with the sampler. Label the bag with sample location information. Typically, collect three EnCore<sup>TM</sup> samplers per sample location. Decontaminate the "T" handle between sample locations.

Using the EnCore<sup>TM</sup> sampler eliminates the need for field preservation and the shipping restrictions associated with preservatives. A complete set of instructions is included with each EnCore<sup>TM</sup> sampler.

After the EnCore<sup>TM</sup> samples are collected, they should be placed on ice immediately and delivered to the laboratory within 48 hours. The samples must be preserved by the laboratory within 48 hours of collection.

#### 2.1.1.2 Soil Samples to be Preserved in the Field

Soil samples preserved in the field may be prepared for analysis using both the low-level (sodium bisulfate preservation) and high- to medium-level (methanol preservation) methods. If samples effervesce when placed in preservative, it is necessary to collect a sample unpreserved, in deionized water. In addition, an unpreserved sample for determination of moisture content must also be collected when collecting soil samples to be preserved in the field.

Methanol Preservation (High to Medium Level). Bottles may be pre-spiked with methanol in the laboratory or prepared in the field. Soil samples to be preserved in the field with methanol shall utilize 40- to 60-milliliter (mL) glass vials with septum-lined lids. Each sample bottle shall be filled with 25 mL of demonstrated analyte-free purge-and-trap grade 3 methanol. The preferred method for adding methanol to the sample bottle is by removing the lid and using a pipette or scaled syringe to add the methanol directly to the bottle.

Soil shall be collected with the use of a decontaminated (or disposable), small-diameter coring device such as a disposable VOA plunger. The outside diameter of the coring device must be smaller than the inside of the sample bottle neck. To collect the sample, pull the plunger back to the required location, insert it into the soil to be sampled, push the coring device into the soil, extrude the soil sample into the methanol-preserved sample bottle, and cap the bottle tightly. Swirl the sample (do not shake) in the methanol to break up the soil such that all of the soil is covered with methanol. Place the sample on ice immediately.

**Sodium Bisulfate Preservation (Low Level).** Bottles may be prepared in the laboratory or in the field with sodium bisulfate solution. Samples to be field-preserved using sodium bisulfate are collected using the same procedures described for methanol preservation.

#### 2.1.2 Procedure for Collecting Soil Samples for Non-VOC Analyses

Samples collected for non-VOC analyses may be collected as either grab or composite samples as follows. Using a sampling device, transfer a portion of soil to be sampled to a stainless steel bowl, disposable inert plastic tray, or baggie. Remove roots, vegetation, sticks, and stones larger than the size of pea gravel. Thoroughly mix the soil to obtain as uniform a texture and color as practicable. Transfer the mixed soil to the appropriate sample containers and close the containers. Place the sample containers immediately on ice.

#### 2.2 TEST PIT AND TRENCH SOIL SAMPLING

Test pit and trench soil sampling is conducted when a complete soil profile is required or as a means of locating visually detectable contamination. This type of sampling provides a detailed description of the soil profile and allows for multiple samples to be collected from specific soil horizons. Prior to conducting any test pit or trench excavation with a backhoe, the sampling team should ensure that the sampling area is clear of utility lines, subsurface pipes, and poles.

A test pit or trench is excavated by incrementally removing soil material with a backhoe bucket. The excavated soil is placed on plastic sheeting well away from the edge of the test pit. A test pit should not be excavated to depths greater than 4 feet unless its walls are properly sloped or stabilized. No personnel shall enter any test pit or trench excavation over 4 feet deep; such action would constitute confined space entry and must conform with Occupational Safety and Health Administration (OSHA) regulations at Title 29 of the *Code of Federal Regulations* § 1910.

Personnel entering the test pit may be exposed to toxic or explosive gases and oxygen deficient environments. Air monitoring is required before entering the test pit, and the use of appropriate respiratory gear and protective clothing is mandatory. At least two persons must be present at the test pit before sampling personnel may enter the excavation and begin soil sampling. Refer to project-specific Health and Safety Plans for required safety procedures for excavations.

Soil samples can also be obtained directly from the backhoe bucket or from the excavated material after it has been removed and deposited on plastic sheeting. The sampling personnel shall direct the backhoe excavator to obtain material from the selected depth and location within the excavation. The backhoe

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operator shall set the backhoe bucket on the ground in a designated location, at a sufficient distance from the excavation to allow the sampler safe access to the bucket. The backhoe operator shall disengage the controls and signal to the sampler that it is safe to approach the bucket. The soil sample shall then be collected from the center of the backhoe bucket to reduce the potential for cross-contamination of the sample.

Test pits are not practical for sampling at depths greater than 15 feet. If soil samples are required from depths greater than 15 feet, samples should be obtained using test borings instead of test pits. Test pits are also usually limited to a few feet below the water table. In some cases, a pumping system may be required to control the water level within the pits.

Access to open test pits should be restricted by the use of flagging, tape, or fencing. If a fence is used, it should be erected at least 6 feet from the perimeter of the test pit. The test pit should be backfilled as soon as possible after sampling is completed.

Various equipment may be used to collect soil samples from the walls or bottom of a test pit. A hand auger, bucket auger, or core sampler can be used to obtain samples from various depths. A trier, trowel, EnCore<sup>TM</sup> sampler, VOA plunger, or spoon can be used to obtain samples from the walls or pit bottom surface.

#### 2.3 SURFACE SOIL SAMPLING

The surface soil sampling equipment presented in this SOP is best suited for sampling to depths of 0 to 6 feet below ground surface (bgs). The sample depth, sample analyses, soil type, and soil moisture will also dictate the most suitable sampling equipment. Prior to sample collection, the sampling locations should be cleared of any surface debris such as twigs, rocks, and litter. The following table presents various surface soil sampling equipment and their effective depth ranges, operating means (manual or power), and sample types collected (disturbed or undisturbed).

Title: Soil Sampling

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Sampling Equipment	Effective Depth Range (feet bgs)	Operating Means	Sample Type
Hand Auger	0 to 6	Manual	Disturbed
Bucket Auger	0 to 4	Power	Disturbed
Core Sampler	0 to 4	Manual or Power	Undisturbed
EnCore <sup>TM</sup> Sampler	Not Applicable	Manual	Disturbed
Spoon/Spatula	0 to 0.5	Manual	Disturbed
Trowel	0 to 1	Manual	Disturbed
VOA Plunger	Not Applicable	Manual	Disturbed

The procedures for using these various types of sampling equipment are discussed below.

#### 2.3.1 Hand Auger

A hand auger equipped with extensions and a "T" handle is used to obtain samples from depths of up to 6 feet bgs. If necessary, a shovel may be used to excavate the topsoil to reach the desired subsoil level. If topsoil is removed, its thickness should be recorded. Samples obtained using a hand auger are disturbed in their collection; determining the exact depth at which samples are obtained is difficult.

The hand auger is screwed into the soil at an angle of 45 to 90 degrees from horizontal. When the entire auger blade has penetrated soil, the auger is removed from the soil by lifting it straight up without turning it, if possible. If the desired sampling depth has not been reached, the soil is removed from the auger and deposited onto plastic sheeting. This procedure is repeated until the desired depth is reached and the soil sample is obtained. The auger is then removed from the boring and the soil sample is collected directly from the auger into an appropriate sample container.

Title: Soil Sampling

2.3.2 **Bucket Auger** 

A bucket auger, equipped similarly as the hand auger, is used to obtain disturbed samples from depths of up to 4 feet bgs. A bucket auger should be used when sampling stony or dense soil that prohibits the use

of a hand-operated core or screw auger. A bucket auger with closed blades is used in soil that cannot

generally be penetrated or retrieved by a core sampler.

The bucket auger is rotated while downward pressure is exerted until the bucket is full. The bucket is

then removed from the boring, the collected soil is placed on plastic sheeting, and this procedure is

repeated until the appropriate depth is reached and a sample is obtained. The bucket is then removed

from the boring and the soil sample is transferred from the bucket to an appropriate sample container.

2.3.3 Core Sampler

A hand-operated core sampler (Figure 1), similarly equipped as the hand auger, is used to obtain samples

from depths of up to 4 feet bgs in uncompacted soil. The core sampler is capable of retrieving

undisturbed soil samples and is appropriate when low concentrations of metals or organics are of concern.

The core sampler should be constructed of stainless steel. A polypropylene core sampler is generally not

suitable for sampling dense soils or sampling at greater depths.

The core sampler is pressed into the soil at an angle of 45 to 90 degrees from horizontal and is rotated

when the desired depth is reached. The core is then removed, and the sample is placed into an appropriate

sample container.

2.3.4 Shovel

A shovel may be used to obtain large quantities of soil that are not readily obtained with a trowel. A

shovel is used when soil samples from depths of up to 6 feet bgs are to be collected by hand excavation; a

tiling spade (sharpshooter) is recommended for excavation and sampling. A standard steel shovel may be

used for excavation; either a stainless steel or polypropylene shovel may be used for sampling. Soil

excavated from above the desired sampling depth should be stockpiled on plastic sheeting. Soil samples

should be collected from the shovel and placed into the sample container using a stainless-steel scoop,

plastic spoon, or other appropriate tool.

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**Trier** 

2.3.5

A trier (Figure 2) is used to sample soil from depths up to 1 foot bgs. A trier should be made of stainless

steel or polypropylene. A chrome-plated steel trier may be suitable when samples are to be analyzed for

organics and heavy metal content is not a concern.

Samples are obtained by inserting the trier into soil at an angle of up to 45 degrees from horizontal. The

trier is rotated to cut a core and is then pulled from the soil being sampled. The sample is then transferred

to an appropriate sample container.

**2.3.6** Trowel

A trowel is used to obtain surface soil samples that do not require excavation beyond a depth of 1 foot. A

trowel may also be used to collect soil subsamples from profiles exposed in test pits. Use of a trowel is

practical when sample volumes of approximately 1 pint (0.5 liter) or less are to be obtained. Excess soil

should be placed on plastic sheeting until sampling is completed. A trowel should be made of stainless

steel or galvanized steel. It can be purchased from a hardware or garden store. Soil samples to be

analyzed for organics should be collected using a stainless steel trowel. Samples may be placed directly

from the trowel into sample containers.

2.4 SUBSURFACE SOIL SAMPLING

Subsurface soil sampling is accomplished in conjunction with borehole drilling, for soil sampling from

depths greater than approximately 6 feet bgs. Subsurface soil sampling is frequently coupled with

exploratory boreholes or monitoring well installation.

Subsurface soil sampling may be conducted using a drilling rig, power auger, or direct-push technology

(DPT). Selection of sampling equipment depends upon geologic conditions and the scope of the

sampling program. Two types of samplers used with machine-driven augers—the split-spoon sampler

and the thin-wall tube sampler—are discussed below. All sampling tools should be cleaned before and

after each use in accordance with SOP No. 002 (General Equipment Decontamination). Both the split-

spoon sampler and the thin-wall tube sampler can be used to collect undisturbed samples from

unconsolidated soils. The procedures for using the split-spoon and thin-wall tube samplers are presented below.

#### 2.4.1 Split-Spoon Sampler

Split-spoon samplers are available in a variety of types and sizes. Site conditions and project needs, such as large sample volume for multiple analyses, determine the specific type of split-spoon sampler to be used. Figure 3 shows a generic split-spoon sampler.

The split-spoon sampler is advanced into the undisturbed soil beneath the bottom of the casing or borehole using a weighted hammer and a drill rod. The relationship between hammer weight, hammer drop, and number of blows required to advance the split-spoon sampler in 6-inch increments indicates the density or consistency of the subsurface soil. After the split-spoon sampler has been driven to its intended depth, it should be removed carefully to avoid loss of sample material. In noncohesive or saturated soil, a catcher or basket should be used to help retain the sample.

After the split-spoon sampler is removed from the casing, it is detached from the drill rod and opened. If VOA samples are to be collected, EnCore<sup>TM</sup> samplers or VOA plungers should be filled with soil taken directly from the split-spoon sampler. Samples for other specific chemical analyses should be taken as soon as the VOA sample has been collected. The remainder of the recovered soil can then be used for visual classification of the sample and containerized for physical analysis. The entire sample (except for the top several inches of possibly disturbed material) is retained for analysis or disposal.

#### 2.4.2 Thin-Wall Tube Sampler

A thin-wall tube sampler, sometimes called the Shelby tube (Figure 4), is used to collect soil samples for geophysical analysis. Tube samplers are best suited for collecting cohesive soils such as clays and silts. The tube sampler may be pressed or driven into soil inside a hollow-stem auger flight, wash bore casing, or uncased borehole. The tube sampler is pressed into the soil, without rotation, to the desired depth or until refusal. If the tube cannot be advanced by pushing, it may be necessary to drive it into the soil without rotation using a hammer and drill rod. The tube sampler is then rotated to collect the sample from the soil and removed from the borehole.

After removal of the tube sampler from the drilling equipment, the tube sampler should be inspected for adequate sample recovery. The sampling procedure should be repeated until an adequate soil core is obtained (if sample material can be retained by the tube sampler). The soil core obtained should be documented in the logbook. Any disturbed soil is removed from each end of the tube sampler. If chemical analysis is required, VOA samples must be collected immediately after the tube sampler is withdrawn. EnCore<sup>TM</sup> samplers or VOA plungers should be filled with soil taken directly from the tube sampler. Before use, and during storage and transport, the tube sampler should be capped with a nonreactive material. For physical sampling parameters, the tube is sealed using plastic caps. The top and bottom of the tube sampler should be labeled and the tube sampler should be stored accordingly.

#### 2.4.3 Direct-Push Technology Sampler

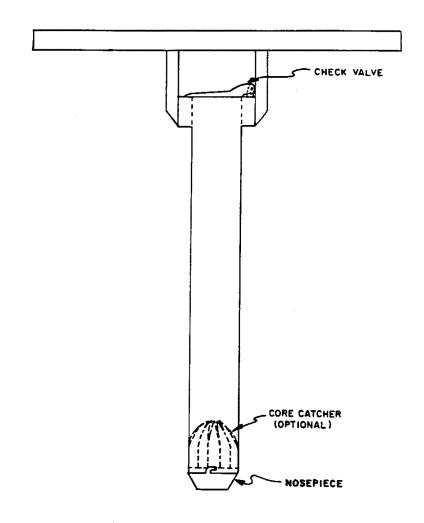
Geoprobe systems utilize DPT. In many cases, DPT is less expensive and faster than collecting soil samples with a standard drilling rig. In addition, the use of DPT causes minimal disturbance to the ground surface and generates little to no soil cuttings. DPT uses acetate or clear polyvinyl chloride (PVC) sleeves for collecting soil samples.

Upon retrieval of the sampling rod from the ground, the sample sleeve is extruded from the sampling rod. The sleeve is sliced lengthwise twice, to open the sleeve. Soil samples can be collected directly from the opened sleeve. If VOA samples are to be collected, EnCore<sup>TM</sup> samplers or VOA plungers should be filled with soil taken directly from the opened DPT sampler. Samples for other specific chemical analyses should be taken after the VOA sample has been collected. The remainder of the recovered soil can then be used for visual classification of the sample and containerized for physical analysis. The entire sample is retained for analysis or disposal.

Title: Soil Sampling

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FIGURE 1
HAND-OPERATED CORE SAMPLER

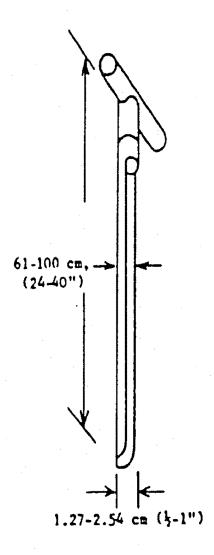


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FIGURE 2

**TRIER** 

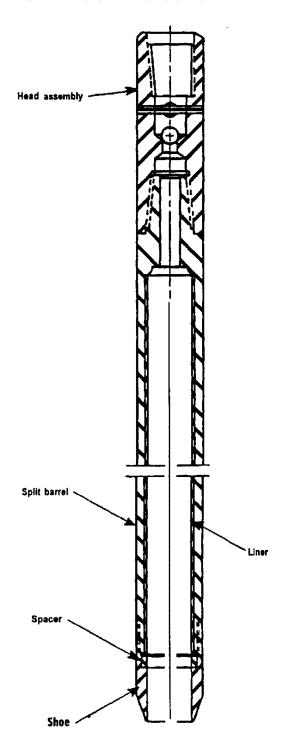
**TRIER** 



Title: Soil Sampling

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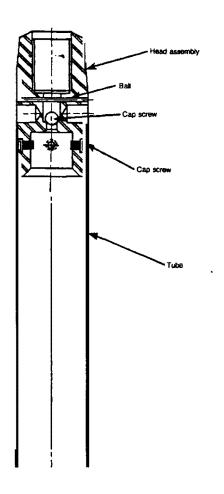
FIGURE 3
GENERIC SPLIT-SPOON SAMPLER



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FIGURE 4 THIN-WALL TUBE SAMPLER



# Region 4 U.S. Environmental Protection Agency Science and Ecosystem Support Division Athens, Georgia

#### OPERATING PROCEDURE

Title:	Field	X-Rav	<b>Fluorescence</b>	Measur	ement
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Effective Date: December 20, 2011 Number: SESDPROC-107-R2

**Authors** 

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### **Revision History**

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
SESDPROC-107-R2, Field X-Ray Fluorescence Measurement, replaces SESDPROC-107-R1	December 20, 2011
General: Corrected any typographical, grammatical and/or editorial errors.	
<b>Title Page:</b> Updated the Enforcement and Investigations Branch Chief to Archie Lee, and the Field Quality Manager to Bobby Lewis.	
<b>Revision History:</b> On the third sentence, replaced Field Quality Manager with Document Control Coordinator.	
<b>Section 1.2:</b> Added the following statement: Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.	
Section 1.3: Revised the last sentence to reflect that the official copy of this procedure resides on the SESD local area network (LAN), and that the Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN.	
<b>Section 1.4:</b> Deleted references to SOPs for old XRF units and added EPA Method 6200 reference.	
Section 1.5.1: Removed two bullets with material related to the R4 SESD Radiation Monitoring Program and old XRF units.	
Section 1.5.2: References to ILS being the only analyst were removed from the second and third sentences.	
<b>Section 1.5.3:</b> On bullet #4, Soil Moisture, removed reference to the use of microwave ovens for sample drying.	
<b>Section 2:</b> A reference to ILS being the only analyst was removed from the $2^{\rm nd}$ paragraph, first sentence. In addition, removed references to SOPs for old instruments and replaced with references to EPA Method 6200 throughout the section.	
Section 3.1: Removed references and associated text for old XRF units and replaced with references to Niton® XLt Series instrument.	
Section 3.2.2: Changed method name from "Cup" to "Collected Sample" Measurement.	

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SESDPROC-107-R1, Field X-Ray Fluorescence Measurement, replaces SESDPROC-107-R0	November 1, 2007
General Updated referenced procedures due to changes in title names and/or to reflect most recent version.	
Title Page Changed title for Antonio Quinones from Environmental Investigations Branch to Enforcement and Investigations Branch.	
Section 1.3 Updated information to reflect that procedure is located on the H: drive of the LAN.	
Section 1.4 Alphabetized and revised the referencing style for consistency.	
Section 2 Added last paragraph regarding stopping measurements due to environmental conditions.	
SESDPROC-107-R0, Field X-Ray Fluorescence, Original Issue	February 05, 2007

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#### 1 General Information

#### 1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when conducting field X-ray fluorescence (XRF) measurements of soil and sediment samples.

#### 1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when measuring metals concentrations in soil, sediment or other solids in the field. On the occasion that SESD field personnel determine that any of the procedures described in this procedure cannot be used to obtain metals analyses of the media being sampled, and that another method or XRF instrument must be used to obtain said measurements, the variant instrument and measurement procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

#### 1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

#### 1.4 References

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

SESD Operating Procedure for Sediment Sampling, SESDPROC-200, Most Recent Version

SESD Operating Procedure for Soil Sampling, SESDPROC-300, Most Recent Version

United States Environmental Protection Agency (US EPA). Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment, Method 6200, Revision 0, February 2007.

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US EPA. 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA.

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version.

#### 1.5 General Precautions

#### 1.5.1 Safety

Proper safety precautions must be observed when conducting field XRF measurements. Refer to the SESD Safety, Health and Environmental Management Program Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. When using this procedure, minimize exposure to potential health hazards through the use of protective clothing, eye wear and gloves. The operator should always be aware of the instrument's radioactive source and the direction of its beam of X-rays. The operator should never point the open source at anyone. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

#### 1.5.2 Procedural Precautions

All field XRF measurements pertinent to the sampling event are recorded in a bound field record logbook for the event. This record is created and maintained by the analyst providing the field XRF support. After the investigation is complete, the analyst will conduct post-processing of the field measurements and will enter final measurement data in the SESD laboratory information management system and provide the SESD project leader with a copy of the field measurement logbook. All other records and documentation of the investigation should be recorded according to the procedures outlined in the SESD Operating Procedure for Logbooks (SESDPROC-010).

#### 1.5.3 Limitations

There are three main sources of interference in XRF analysis that may impact data quality. They are sample preparation error, spectral interferences and chemical matrix interferences.

• Preparation Error – The accuracy of the analysis is strongly impacted by sample homogenization. The more homogeneous the sample, typically analyzed by the cup method, the more accurate the results. There is no control of this limitation when conducting in situ analysis.

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- Spectral Interference Each element has a signature spectrum of energies and relative intensities. Many elements, however, produce X-rays of similar energy and discerning which element produced a detected X-ray is a factor of the detector's resolution capability and the software's ability to fit all of the data to the relative intensities produced by the various wavelengths.
- Chemical Matrix Interference This refers to the effect that one element has on another in producing X-rays which reach the detector. Dominant elemental components of a sample, such as silicon in soils, vary in concentration from sample to sample and therefore so does that element's influence on the other elements in the sample.

There are several other limitations that the field investigator must be acutely aware of when conducting field analysis using XRF.

- Soil moisture Excessive soil moisture biases the results low, i.e., the higher the soil moisture in a particular matrix, the lower the reported concentration relative to the actual concentration. This limitation may be overcome by drying the sample. Without sample drying, XRF measurement results for samples with typical soil moistures within the range of 15% 25% are routinely reported at values less than laboratory confirmation analysis for the same samples. The actual difference may vary significantly for all samples from a site but the XRF results reported by the instrument are typically on the order of 70% 80% of the laboratory reported value for samples in this moisture range. This factor should be taken into consideration when making decisions based on XRF results.
- Lack of sensitivity with respect to certain analytes Due to peak overlaps, some analytes may have problematically high detection limits, i.e., detection limits may be higher than project action levels for certain analytes, limiting its use for rapid field screening for certain elements. One of the most common examples of this phenomenon is the lead/arsenic analyte pair. When lead and arsenic are being analyzed, the peak overlap problem results in detection limits for arsenic that are several times higher than the typical action levels published for this analyte. It commonly is necessary to perform confirmatory analysis in the laboratory to obtain analytical results for arsenic, or other analytes with high detection limits, to obtain data in the range necessary for making regulatory decisions.

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#### 2 Operational Checks and Quality Control

All XRF instruments shall be maintained and operated in accordance with the manufacturer's instructions, EPA Method 6200 and the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). Prior to each operational period, the instrument is turned on and is allowed to perform an internal calibration. Following this calibration, a performance check is conducted, using the appropriate National Institute of Standards and Technology (NIST)-traceable standard reference material for the analytes of concern. The value should be within +/- 20% of the stated value of the standard. Following this performance check, an instrument blank sample is analyzed to verify the instrument is not registering false positive results for the analytes of concern. After these checks, the instrument is ready for analysis.

The following operational and quality control requirements also apply to operation of the XRF instrument and must be followed and documented in the field logbook maintained by the analyst:

- During operations, the ambient air temperature will be recorded for each measurement and if the ambient temperature changes by more than 10°F, the instrument will be recalibrated.
- A method blank is analyzed at least once a day to determine if contamination is entering the analytical procedure.
- While the instrument is being used, the reference standards and the blank are run once each hour or every twenty samples, whichever occurs first, and also at the end of the period of operation, prior to turning the instrument off.
- For every twenty samples, or at least once per day, analyze a duplicate using the main sampling technique.
- Once per day, check the instrument's precision by analyzing one of the site samples at least seven times in replicate.

EPA Method 6200 contains detailed instruction and guidance covering implementation of these procedures and any corrective actions that must be taken based on measured instrument behavior and performance. If at any time during a field investigation, it appears that the environmental conditions could jeopardize the quality of the measurement results, the measurements will be stopped. This will be documented in the field logbook.

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#### **3** Field X-Ray Fluorescence (XRF) Measurement Procedures

#### 3.1 General

XRF is the property of a material to emit X-rays, with a characteristic energy, upon being irradiated by X-rays from a known radioisotope source. The emitted X-rays are detected by the particular XRF instrument as they impact a detector, which converts the energy of the emitted X-ray into electric current. The strength of the current is proportional to the energy of the X-ray. An onboard microprocessor counts how often an energy is detected, assigns the energy to a particular element and reports the calculated concentration for the element.

The XRF instrument available for use by SESD field investigators is the Niton® XLt 700 Series Multi-element XRF Spectrum Analyzer. This instrument uses a miniaturized X-ray tube as its source rather than a radioactive isotope for X-ray generation for analysis.

#### 3.2 Mode of Operation

The instrument is typically used in one of two modes, either for taking in situ measurements or for measuring sample material that has been placed in a cup for analysis in the instrument tray. The following is a brief description of these modes of operation.

#### 3.2.1 In Situ Measurement

Prior to taking the in situ measurement, the measurement location is cleared of any significant vegetation, such as large clumps of grass, and is scuffed or otherwise leveled to provide a flat surface on which to place the instrument window. A piece of thin Mylar® film is then placed on the measurement location. This protects the instrument window, preventing it from becoming damaged or contaminated by the media being tested. After the window is pressed to the Mylar® film, the window is opened for a nominal (i.e., programmed) sixty seconds.

Because of the shallow penetration of the X-rays in typical soils, the measured concentrations are representative of the concentrations present at the very surface of the material being measured. If conditions representing concentrations over a greater depth are required by the study data quality objectives (i.e., on the order of three to six inches), the cup method, described in Section 3.2.2, must be used.

#### 3.2.2 Collected Sample Measurement

This method is used to measure concentrations of metals in soil and sediment samples collected from a vertical interval, either as a grab or a composite sample. Typically, soil or sediment samples are collected as if the samples were being collected for routine chemical analyses (SESD Operating Procedure for Soil Sampling (SESDPROC-300) and the SESD Operating Procedure for Sediment

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Sampling (SESDPROC-200). After mixing, the media being sampled may be placed in either an 8-ounce glass container or a clean, unused zip-closure plastic bag (or equivalent). The XRF analyst then takes an aliquot from the container and places it in a small plastic cup with a Mylar® covering. The cup containing the sample is then loaded into a tray for analysis by the XRF instrument. Alternatively, if project objectives allow, measurements may be obtained by reading directly through the plastic bag. Window opening time considerations are the same as for the in situ measurement procedures described in Section 3.2.1.

The concentrations reported for the samples analyzed by the cup method are representative of the interval sampled, i.e., if the sampler collected the sample from the interval of 0 to 3 inches below ground surface, the reported concentration, assuming thorough homogenization, will be an average of the concentrations over that interval.

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#### 4 Study Design

#### 4.1 General

XRF instruments are typically used for two main purposes. First it may be used to rapidly assess site conditions to support a site reconnaissance. Secondly, it may be used to screen large numbers of soil or sediment samples to minimize the number of samples that are sent to a laboratory to provide detailed site characterization data. These uses are summarized in the following sections.

#### 4.2 Reconnaissance

XRF may be used to obtain in situ measurements at a large number of locations in a short period of time to determine if a site warrants further attention with respect to characterization. When used in concert with GPS, and when observing the limitations described in Section 1.5.3, XRF can reveal, where present, contamination patterns at a site which can form the basis for development of a more detailed study to provide definitive data for site characterization. Conversely, the reconnaissance results may form the basis for a "no further action" decision, providing a very cost effective tool for the decision maker.

#### 4.3 Screening Support for Definitive Level Site Characterization

XRF may be used to supplement laboratory analyses to allow for the collection of large numbers of samples to provide a detailed characterization of a site. A high sample density grid or sampling pattern is created to provide adequate detail to meet the data quality objectives of the study or investigation. This sampling pattern may also involve the collection of significant numbers of subsurface soil samples to characterize any contamination present in the subsurface.

All samples, collected according to procedures found in SESD Operating Procedure for Soil Sampling (SESDPROC-300) and SESD Operating Procedure for Sediment Sampling (SESDPROC-200), are delivered to the XRF analyst on site. The analysis of these samples is conducted according to the method described in Section 3.2.2 of this procedure.

Based on the limiting factors described in Section 1.5.3, a confirmatory analytical scheme can be developed which minimizes the numbers of samples that must undergo laboratory analyses, yet provides definitive level data, with a high degree of confidence, to the project leader and other decision makers. Using the moisture limiting factor, there is usually a high degree of confidence that samples that screen at concentrations less than approximately 70% - 80% of the site action level will actually exceed the action level. Of the samples that screen at the action level, most all will, with a high degree of confidence, exceed the action level. If a reconnaissance is conducted prior to the full-scale site investigation, in addition to the *in situ* analysis, it is advisable to collect and

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analyze a small subset of the screened locations to generate site-specific moisture limiting factors. This correlation factor can be used to develop a sampling scheme with more confidence.

Using these relationships, the following scheme may be implemented:

- Ten percent of the samples that screen at concentrations less than approximately 70% 80% (or other correlation factor developed on actual data) of the site action levels are submitted for confirmation analyses to confirm that concentrations are, in fact, below the site action levels.
- All of the samples that screen at concentrations of 70% 80% (or other correlation factor developed on actual data) of the action level up to the action level value are submitted for confirmation analyses to confirm that concentrations are, in fact, equal to or greater than the site action levels.
- Ten percent of the samples that screen at concentrations exceeding the action levels are submitted for confirmation analyses to confirm that concentrations are, in fact, greater than the site action levels.